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PROCESSING STUDIES OF
ALUMINUM-MAGNESIUM AND
ALUMINUM-COPPER-LITHIUM ALLOYS

by

Frank J. Harsacky Jr.

March 1990

Thesis Advisor

T.R. McNelley

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Processing Studies of Aluminum-Magnesium and
Aluminum-Copper-Lithium Alloys

by

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ABSTRACT

Investigation into the effect on superplastic behavior of two aluminum alloys produced by variations of thermomechanical processing parameters was conducted. The alloys in this study are Al-10Mg-0.1Zr (weight percent) and 2090, which is Al-2.56Cu-2.03Li-0.12Zr (weight percent). Determination of the existence of an optimum balance between deformation and recovery for the Al-10Mg-0.1Zr alloy was accomplished by extending the annealing interval to 60 minutes during warm rolling at 300°C. The optimum balance is a 30 minute annealing interval between rolling passes. Processing of Al-10Mg-0.1Zr with a rolling temperature lower than the annealing temperature produced ductilities which are less than those obtained by utilization of the optimum process. The extension of annealing intervals in the processing of 2090 resulted in increased superplastic response when compared with results obtained employing shorter annealing intervals. By application of a two-temperature process which incorporates rolling at a lower temperature than the annealing temperature, the determination has been made that enhanced ductility results however, the annealing interval of 15 minutes should be extended.



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I. INTRODUCTION

The demands of reducing the cost and complexity of aircraft components while improving strength, durability and other material characteristics have resulted in substantial research in the field of superplastic metals. A superplastic material is one that is able to undergo tensile elongations of 200 percent or greater without local necking. This particular attribute enables fabrication of one-piece components which heretofore had to be manufactured as an assembly of multiple parts. This fabrication technique is superplastic forming and its attractive features include elimination of fasteners and the associated stress concentration caused by fastener holes which result in both fatigue and crevice corrosion. Economic benefits result from reduced labor costs at the component assembly level, which translate into lower acquisition costs as well as a reduction of life cycle costs due to fewer parts to maintain.

Airframes and other aircraft structural members are constructed of aluminum and thus much of the research in superplastic behavior has considered aluminum alloys. Superplastic elongations with high strength aluminum alloys such as 7475 have been achieved, but because of the high temperatures involved in the process (500°C ; $> 0.8T_m$) cavitation occurs, resulting in a loss of strength [Refs. 1, 2]. Control of cavitation requires application of back pressure which complicates the process and restricts somewhat the size of formed components. In an effort to produce superplastic behavior in high strength aluminum without cavitation the Naval Postgraduate School (NPS), with the support of Naval Air Systems Command (Navair), began research in processing techniques for superplasticity in aluminum alloys. A thermomechanical process (TMP) was developed which utilized hot forging followed by warm rolling with reheating or annealing intervals between the rolling passes. Tensile testing of an Al-Mg alloy processed with this TMP technique has demonstrated superplastic elongations at relative low temperatures (300°C) and high strain rates. In addition, little cavitation is seen during such low temperature superplastic flow. Since then there have been numerous studies of many different aluminum alloys and investigation into the effects on the microstructure and mechanical properties of varying the time and temperature parameters of the TMP and testing procedures. Extensive study has been conducted on the microstructures in order to point the way for further research, and to provide a better understanding of the mechanism of microstructural refinement during processing.

Most recently, Gorsuch has investigated the effects of varying the annealing time between rolling passes, and found that by the incorporation of a 30 minute annealing interval in the TMP, tensile elongations in excess of 1000 percent could be obtained during testing at 300°C [Ref. 3]. A portion of this research will follow that of Gorsuch by extending the annealing interval to 60 minutes in order to determine whether the 30 minute interval is at or near the optimum annealing time for superplastic processing.

Much of what has been learned from this Al-Mg research can be applied to other aluminum alloys. More specifically, research at NPS in recent years has included studies with the high strength aluminum alloys 2090 and Navalite. Although these higher strength alloys do not exhibit the spectacular superplasticity of the Al-Mg alloys when processed by the methods applied to the Al-10Mg materials, they do show promise. Results to date include superplastic tensile elongations when processed with a modified TMP similar to that developed for the Al-Mg alloys. The Al-Mg TMP has been used as a model to guide the research efforts with other alloys, and to predict the expected results. This research has also applied the techniques developed for Al-Mg processing to that of 2090, in a continuation of research with that alloy, which began with Spiropoulos in 1987 [Ref. 4]. A portion of this research follows the more recent work of Reedy, who in 1989 studied the effects of variation of rolling temperature on the resultant superplastic tensile elongation [Ref. 5]. This current research investigates the variation of annealing intervals and also of employing different temperatures for the rolling and annealing. These variations in TMP were also applied to experimentation with Al-10Mg in order to observe the effect on the superplasticity obtained in comparison with the previous results, and to gain insight which might be utilized to further 2090 research. This thesis reports on the status of current research at NPS on these developments and specifically considers the variations to the TMP utilized in superplastic processing of Al-10Mg and 2090 Aluminum.

II. BACKGROUND

The commercial production of aluminum on a large scale dates its origins back one hundred years to the discovery of the Hall-Heroult process for reduction of ore to metal [Ref. 6]. The demand for a strong and lightweight metallic material provided steady growth for the industry. However, it was the inception of the aircraft industry that eventually spurred the rapid development of aluminum alloys and dramatic expansion for the industry.

The widespread use of aluminum in aircraft began with lightweight cast aluminum alloy engine pistons. There were more than 250,000 aircraft engines produced during World War I and this resulted in a huge increase in demand for aluminum and its alloys [Ref. 7]. Since that time, demands of the aircraft industry, coupled later with those of the aerospace industry, have been responsible for much of the research leading to lighter and stronger aluminum alloys. Along with the search for better alloys has come the need for improved methods of fabrication. One method which has undergone substantial development in recent years is superplastic forming (SPF). Aluminum alloys must be prepared by a TMP in order to achieve superplastic capability. Then, SPF utilizes the material's superplastic elongation to produce a component in a manner similar to vacuum forming. To date, the components fabricated by this process are used in non-structural and secondary structural members due to cavitation and related microstructural defects which often result in diminished service performance. In order to produce superplastically formed components of high strength and mechanical integrity, a different TMP may be required. A two-stage TMP has been developed which produces superplastic tensile elongations at a relatively low temperature (300°C) and without cavitation in Al-Mg alloys. This initial research experimented with an Al-10Mg-0.1Zr (wt. pct.) alloy. Figure 1 is a schematic illustration of the TMP developed.

The first stage of the TMP consists of solution treatment and hot working by upset forging. This is conducted at a temperature above that of the solvus but below the eutectic, to produce a homogeneous solid solution. The second and most critical stage of this TMP is the warm rolling. This is accomplished at a sufficiently low temperature to prevent recrystallization via nucleation and grain growth, but at a temperature high enough that both the recovery and precipitation of the β (Al_3Mg_2) phase can take place.

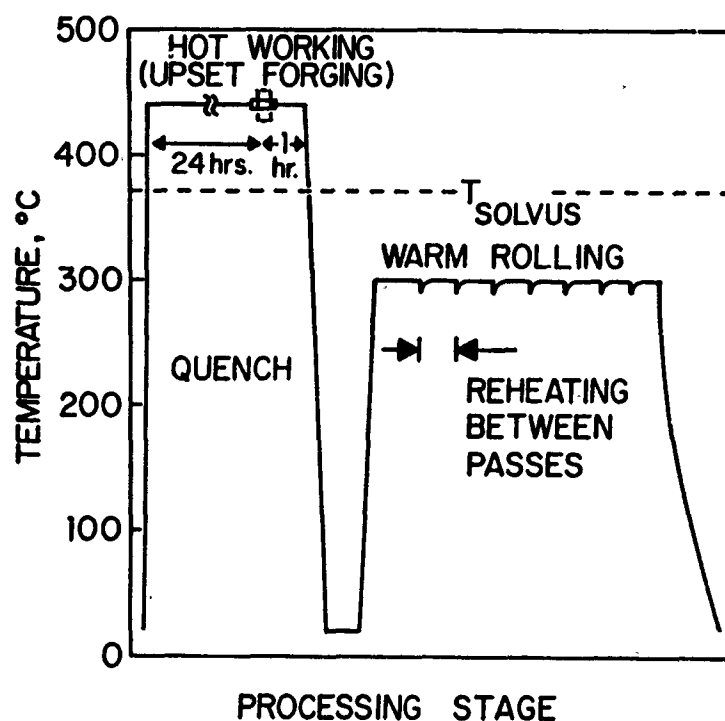


Figure 1. Thermomechanical Process

During the warm rolling process a rolling pass introduces a dislocation structure. Upon reheating and annealing, for a period of time, recovery of the dislocation structure will occur with formation of a cellular or subgrain structure. Precipitation of the β -phase occurs upon the nodes of the subgrain structure, and tends to stabilize it so that, upon subsequent deformation and annealing cycles, more dislocations are introduced and recovered to the pre-existing subgrain structure. By means of this alternating deformation and annealing, a build up of boundaries of sufficient misorientation occurs to constitute a recrystallized grain structure [Ref. 8].

An important observation made in the case of Al-10Mg-0.1Zr is that a proper balance between strain induced per rolling pass and the annealing time can produce an extremely high superplastic ductility in subsequent stress-strain testing. This has led to the most recent research on an Al-10Mg-0.1Zr(wt.pct) alloy wherein Gorsuch varied the annealing times between rolling passes. As the annealing times are increased from 5 minutes to 12.5 minutes and on up to 30 minutes, ductility in subsequent testing is progressively increased [Ref. 9].

A series of research projects have been conducted at NPS utilizing various aluminum alloys including 7475 [Ref. 2], NAVALITE [Ref. 10], and 2090 [Refs. 11,12,5, 13]. In the case of 2090, an Aluminum - Copper - Lithium alloy, an attempt was made to achieve a similar microstructure by TMP of the alloy in a manner similar to the methods developed for Al-Mg alloys.

Research with 2090 at NPS began with that of Spiropoulos in 1987 [Ref. 11]. Groh extended this work and investigated whether the non-coherent, equilibrium T_1 and T_2 phases in 2090 would act in a similar manner to that of the β -phase in the Al-Mg alloys. The results of Groh's research showed that the T_2 phase does in fact behave like the β -phase in Al-Mg alloys, in that the T_2 phase precipitate has a stabilizing effect on the evolving subgrain structure during warm rolling, allowing subsequent deformation and annealing cycles to produce fine structures. The T_1 morphology essentially stayed the same during rolling, and did not appear to be affected by the deformation. In fact, the results indicate that T_2 replaced T_1 during the processing [Ref. 14].

Choudhry and Reedy [Refs. 5,13], conducted subsequent work with the processing by experimentation with balancing the deformation strain with the annealing times in an effort to produce high superplastic ductilities similar to those which resulted from the Al-10Mg research. The results weren't noteworthy in terms of ductility produced when compared with those of the Al-Mg research. However, moderate superplastic ductilities produced at relatively low temperatures did result from this research. The low temperatures and strain rates employed in these experiments were similar to those utilized in the high magnesium aluminum alloys. These results, coupled with those of the earlier 2090 research indicate that a balance between the deformation strains and annealing times may exist for the processing of this alloy to achieve superplastic response at lower temperatures and in a manner similar to the Al-Mg materials.

This thesis begins with an extension of Gorsuch's work on annealing times during processing of the Al-10Mg-0.1Zr alloy. In following the model that has been developed for Al-Mg alloys over the years, as annealing times are extended, grain growth and a coarsening of the sub structure is expected. The subject of the first portion of this research is to determine where this grain coarsening and the ensuing loss of ductility in subsequent testing occurs.

Reedy's research results also indicated that enhanced ductility might result from extending annealing times in processing of 2090. Following Reedy, the second portion of this research extends the annealing time in processing of the 2090. It was also demonstrated that a decrease in rolling temperature produced an increase in ductility. The

final portion of this research investigates the effects of conducting the rolling deformation at temperatures lower than that of the annealing intervals. The two-temperature rolling scheme was applied to both alloys: it was utilized with the Al-Mg material to investigate how the model alloy would react to the variation in the TMP and whether ductility enhancement would result. The two temperature process was then applied to the 2090 in a effort to balance strain history with annealing history to attempt to accomplish a refined, continuously recrystallized structure at the conclusion of the rolling process such that superplastic ductility at relatively low temperatures would be obtained.

III. EXPERIMENTAL PROCEDURE

A. MATERIAL

1. Al-10Mg-0.1Zr

The composition of the aluminum-magnesium alloy utilized in this research is contained in Table 1.

Table 1. COMPOSITION OF ALLOY AL-MG (WEIGHT PERCENT)

Magnesium (Mg)	Zirconium (Zr)	Aluminum (Al)
9.89	0.09	Balance

The material was provided as a direct-chill cast ingot measuring 150 mm diameter x 580 mm length (6 in diameter x 23 in length). This is casting number S572826, provided by the ALCOA Technical Center, ALCOA Center, Pennsylvania.

2. 2090

The aluminum alloy 2090 studied in this research was received in rolled plate form, with measurements of 510 mm length x 318 mm width x 38 mm thickness (20 in length x 12.5 in width x 1.5 in thickness). The composition is given in Table 2. The plate was heat treated to a T8E41 temper (as received condition).

Table 2. COMPOSITION OF ALLOY 2090 (WEIGHT PERCENT)

Copper (Cu)	Lithium (Li)	Zirconium (Zr)	Aluminum (Al)
2.56	2.03	0.12	Balance

B. PROCESSING

Thermomechanical processing techniques used in this research follow those developed during previous NPS studies of Al-10Mg alloys [Ref. 9] and 2090 [Ref. 13]. The process variations utilized in this research are detailed below.

1. Sectioning and Billet Preparation

Part of the Al-10Mg-0.1Zr casting had been sectioned into small billets with dimensions of 95 mm x 31 mm x 31 mm (3.75 in x 1.25 in x 1.25 in). The actual billet sectioning had been accomplished in previous work [Ref. 9]. The 2090 billets were also sectioned from the rolled plate in previous work [Ref. 13]. The measurements were 55 mm in the rolling direction x 43 mm wide x 43 mm thick.

2. Solution Treatment and Forging

Solution treatment for both alloys was conducted in a Lindberg type B-6 Heavy Duty furnace. A Baldwin-Tate-Emery Universal Testing machine was used to accomplish the upset forging of both alloys. The Al-10Mg-0.1Zr billets were solution treated for 24 hours at 440°C for homogenization. After the initial solution treatment, the billets were forged along the longitudinal direction to a thickness of 25.4 mm (1 in) between platens heated to 440°C. The billets were then returned to the furnace for one more hour of solution treatment, after which they were quenched in water.

The 2090 billets were solution treated for sixteen hours at 540°C to homogenize the material. This temperature is below the Al_3Zr solvus and above the T_1 and T_2 solvus temperatures. Upon completion of the initial solution treatment, the billets were forged between platens heated to their maximum temperature of 480°C. The billets were forged along their short transverse direction to a thickness of 25.4 mm (1 in). After forging, the billets were returned to the furnace for further solution treatment of two additional hours, upon completion of which the billets were quenched in water.

3. Rolling

The rolling performed on all billets was accomplished with a Fenn Laboratory Rolling Mill. The 111 mm (4.3 in) diameter rolls opened to maximum of 25.4 mm (1 in). As the rolls and entry table are not heated, it was imperative that the billets be transferred from the annealing furnace to and through the rolls in a timely manner in order to maintain an approximately isothermal condition. Rolling and material transfer techniques were developed to satisfy this requirement.

a. Al-10Mg-0.1Zr

There were three separate TMP's utilized with this alloy. The same rolling schedule was followed for all rolling performed in this research, for both alloys, and is contained in Table 3.

Table 3. ROLLING SCHEDULE

ROLL #	ROLL CHANGE (0.08in + 0.01in)	MILL SETTING (right/left)	MILL GAP (in)	MILL GAP (mm)
1	+(12 + 4)	0/0	1.00	25.4
2	-(2 + 0)	0/0	0.84	21.3
3	-(1 + 2)	6/6	0.74	18.8
4	-(1 + 2)	4/4	0.64	16.3
5	-(1 + 2)	2/2	0.54	13.7
6	-(1 + 2)	0/0	0.44	11.2
7	-(1 + 2)	6/6	0.34	8.6
8	-(1 + 2)	4/4	0.24	6.1
9	-(0 + 6)	6/6	0.18	4.6
10	-(0 + 6)	0/0	0.12	3.05
11	-(0 + 5)	3/3	0.07	1.78
12	-(0 + 2.2)	0.8/0.8	.048	1.22

The first, TMP A, employed a 60 minute annealing interval at 300°C between rolling passes. Figure 2 is a schematic of TMP A. This follows previous research where annealing times of 5, 12.5 and 30 minutes were studied [Ref. 9]. A 60 minute annealing interval rolling schedule was studied in this research to determine whether a recovery time existed beyond which there is no improvement in the superplastic response.

The second and third rolling schedule, TMP B and TMP C (Figures 3 and 4) employed in this study involved varying the rolling temperature from the annealing temperature. This was done to investigate whether ductility improvement would result from a reduced rolling temperature.

In the first of these varied temperature rolling schedules, TMP B, the alloy was annealed at 300°C for a period of 30 minutes after each of the first five rolling passes. Beginning with rolling pass number six, the alloy was again annealed for 30 minutes at 300°C, after which the alloy was transferred to another furnace set at 250°C. The material was held at this lower temperature for 15 minutes to allow for temperature stabilization and then rolling was accomplished. This two tier temperature system was used for the remainder of the rolling passes.

The second varied temperature rolling schedule was identical to the first with the following exception: after the fifth rolling pass the alloy was held at 300°C for 15 minutes (vice 30 minutes) and then cooled to 250°C for 15 minutes.

For all three rolling schedules, the rolled sheets were water quenched upon completion of the final rolling pass.

b. 2090

Four TMP's were utilized in the 2090 alloy processing. The first one, TMP D (Figure 5) is a 60 minute annealing interval identical to the Al-10Mg-0.1Zr 60 minute anneal rolling schedule. The remaining three were of the variable temperature type, similar to those used in the Al-10Mg-0.1Zr study. These three each had a 30 minute annealing period at 300°C for the first five rolling passes. Beginning with rolling pass number six, the annealing time was reduced to 15 minutes, after which the alloy was transferred to another furnace set at the rolling temperature. The alloy was held in the furnace for 15 minutes to permit complete temperature stabilization. The three TMP's utilized, with their corresponding rolling temperatures are: TMP E, 250°C; TMP F, 200°C; and TMP G, 150°C (Figures 6,7 and 8). For all four TMP's, the rolled sheets were water quenched upon completion of the final rolling pass.

4. Sample Preparation

Tensile test samples were sectioned from the rolled material. They were machined to dimensions for tensile testing as shown in Figure 9. The tensile test samples were produced such that the test axis was parallel to the rolling direction.

C. TESTING

Testing of the Al-10Mg-0.1Zr alloy was performed on both an Instron Model TT-D Testing machine fitted with a Marshall three zone clam shell furnace and an Instron Model 6027 testing machine equipped with an Astro tubular furnace. The 2090 alloy samples were tested on the Instron Model 6027 testing machine with the Astro tubular furnace.

The Instron TT-D produced a strip chart with load vs. time recorded, whereas the Instron 6027 output was corresponding values of load, time and position. The Al-10Mg-0.1Zr samples were tested at 300°C at strain rates ranging from $6.67 \times 10^{-5} \text{ sec}^{-1}$ to $6.67 \times 10^{-2} \text{ sec}^{-1}$. The 2090 samples were tested at temperatures of 350°C, 400°C, 425°C and 450°C at three strain rates; $6.67 \times 10^{-5} \text{ sec}^{-1}$, $6.67 \times 10^{-4} \text{ sec}^{-1}$ and $6.67 \times 10^{-3} \text{ sec}^{-1}$.

D. DATA REDUCTION

Data output from the Instron testing machines was corrected to compensate for the decrease in true strain rate with the increasing strain. The compensation method details have been previously outlined [Ref. 15]. Engineering stress vs. engineering strain curves and true stress vs. true strain curves were produced for both alloys from the testing results. Ductility vs. strain rate curves were produced for the Al-10Mg testing. Ductility vs. tensile test temperature curves were produced for the 2090 alloy testing. Flow stress (true stress) at a 10 percent strain vs. strain rate curves were developed for the Al-10Mg-0.1Zr and 2090 alloys. In addition, flow stress (true stress) at a 10 percent strain vs. tensile test temperature were produced for the 2090 alloy.

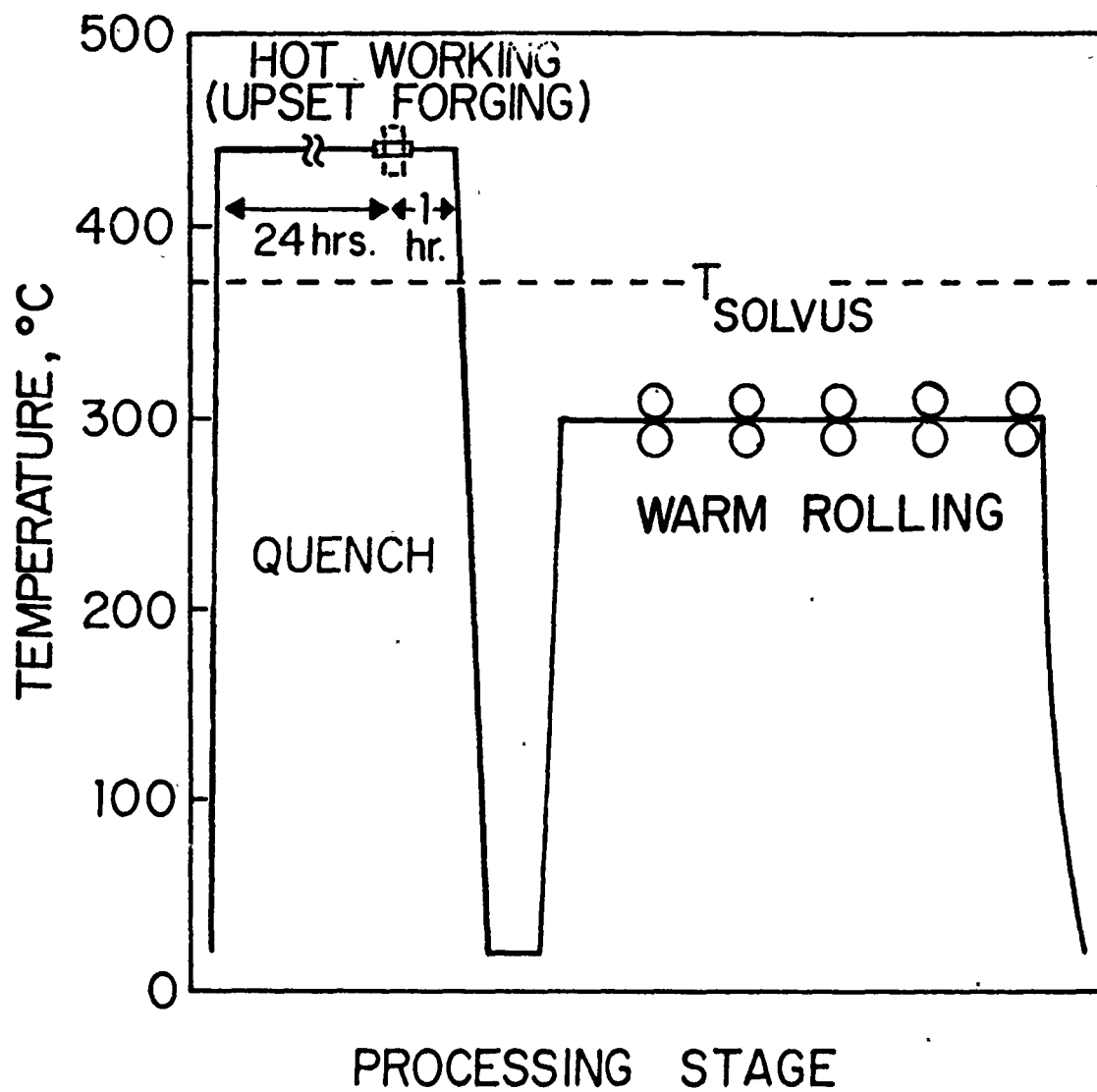


Figure 2. TMP A

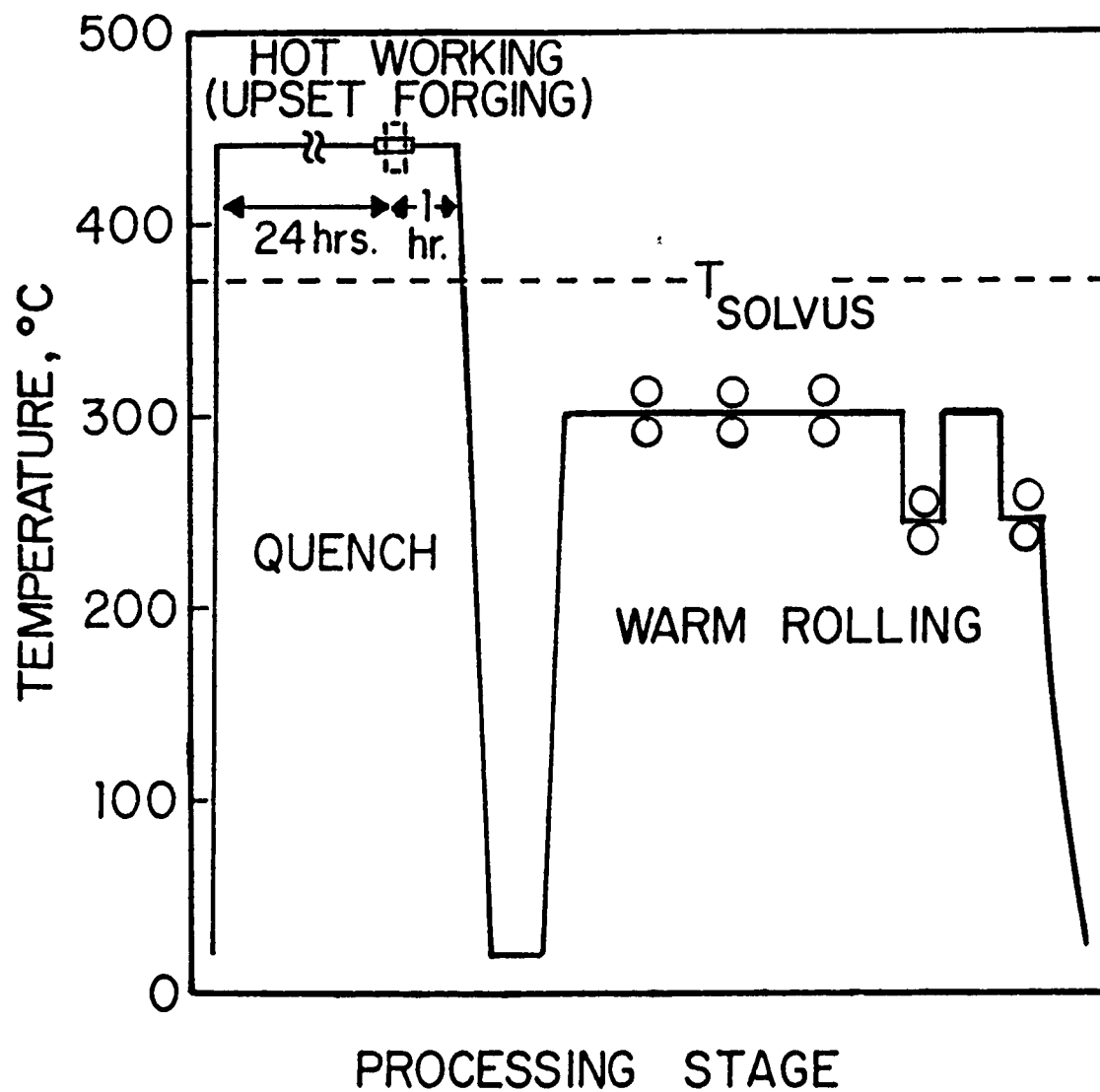


Figure 3. TMP B

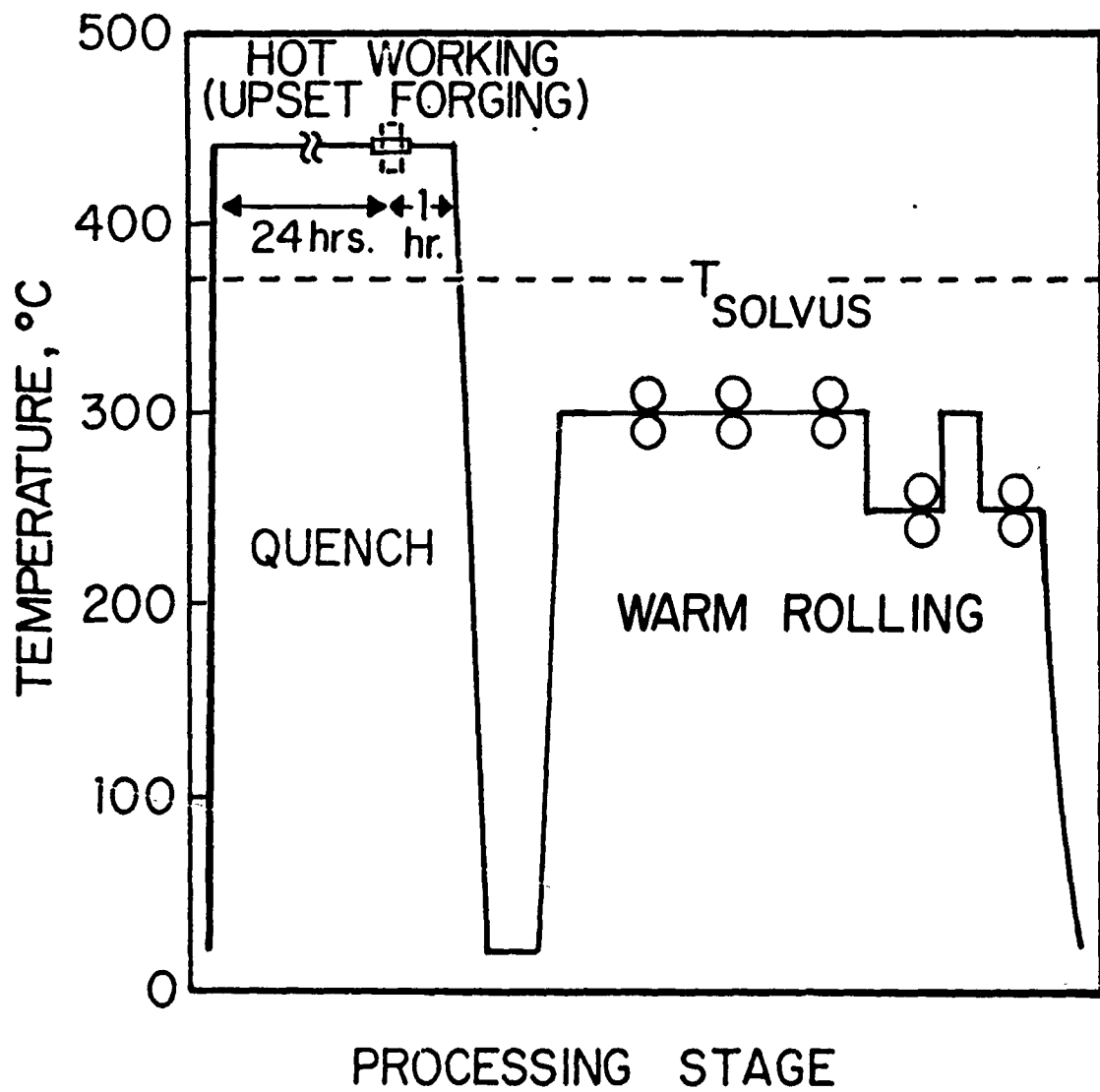


Figure 4. TMP C

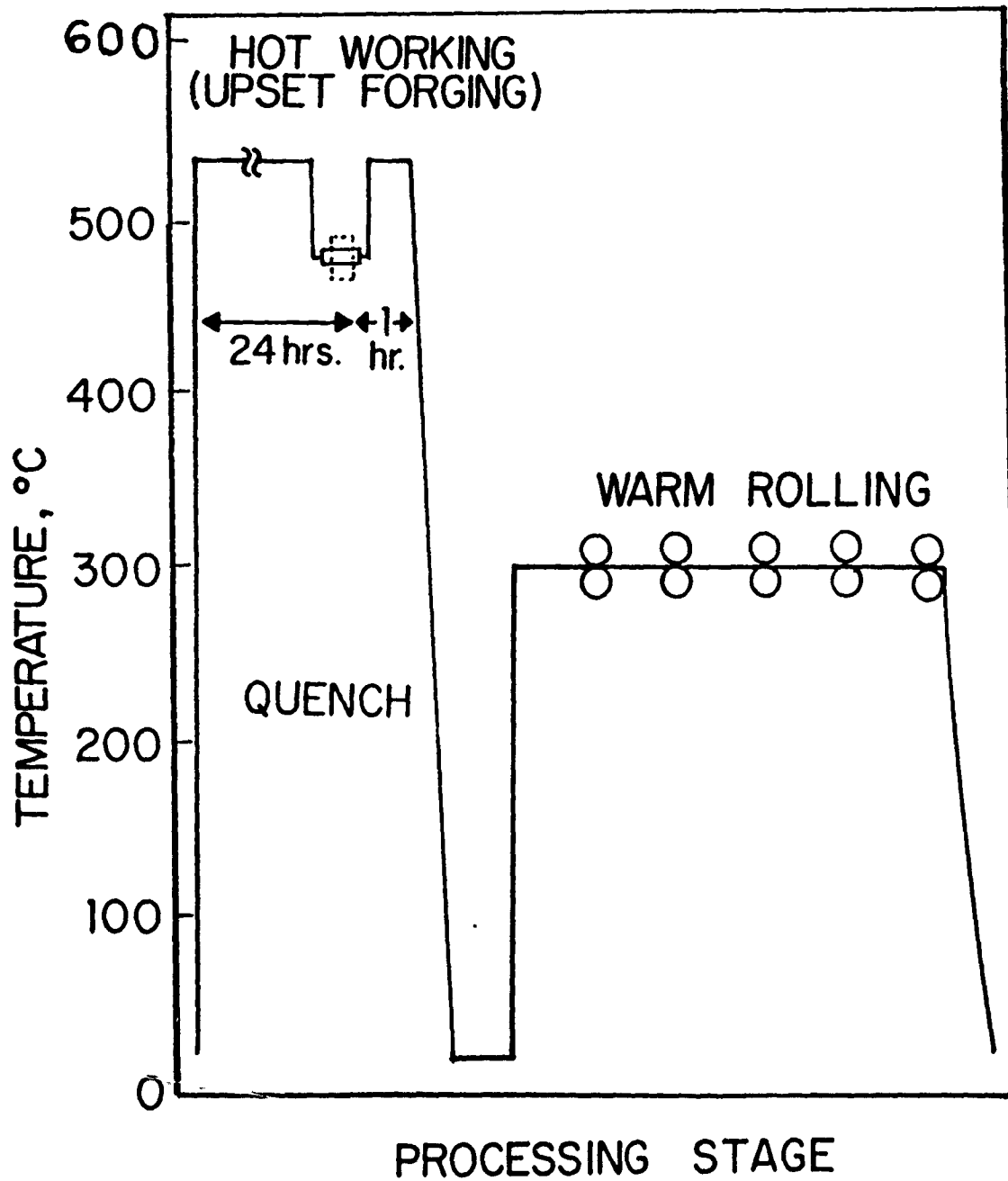


Figure 5. TMP D

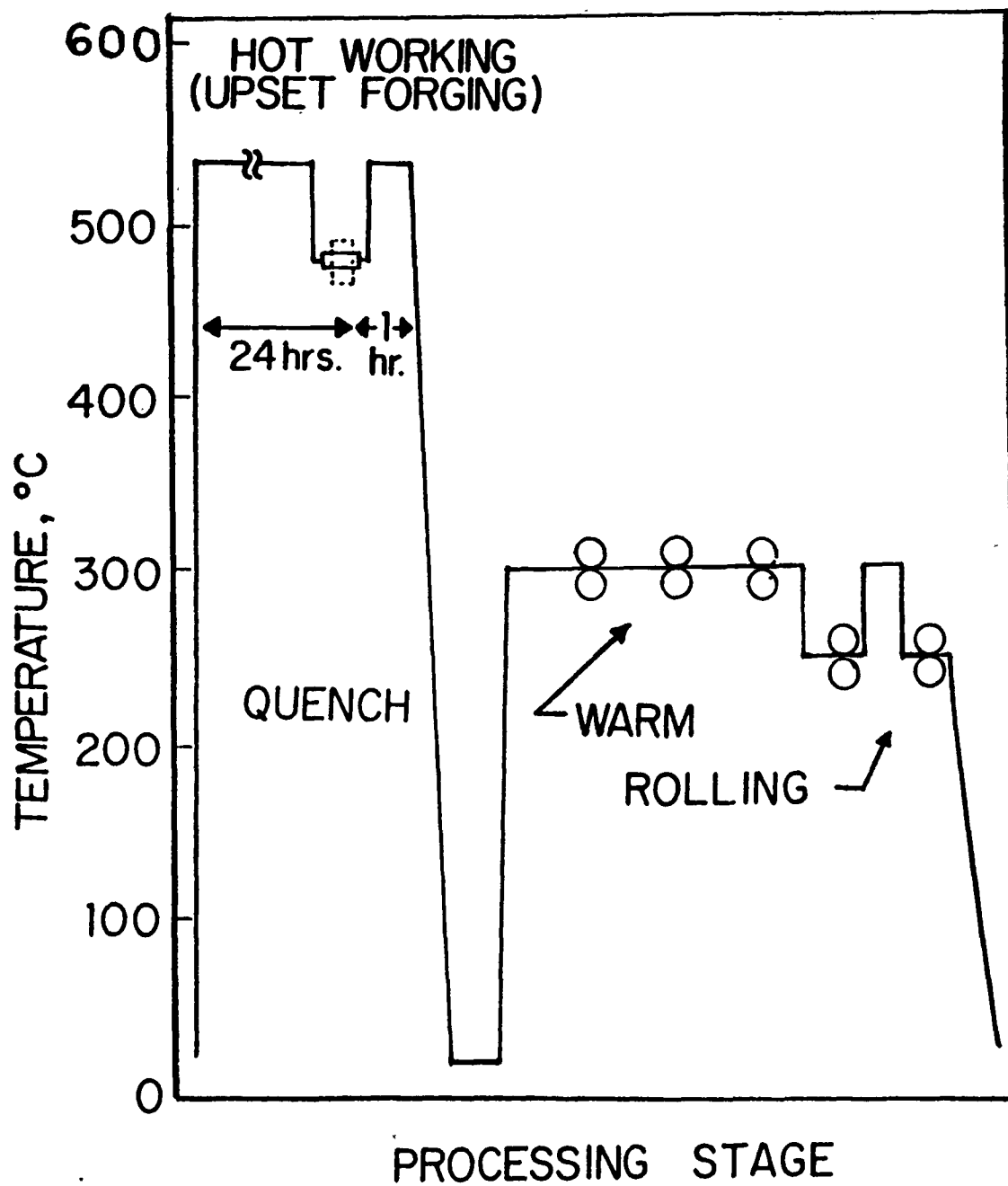


Figure 6. TMP E

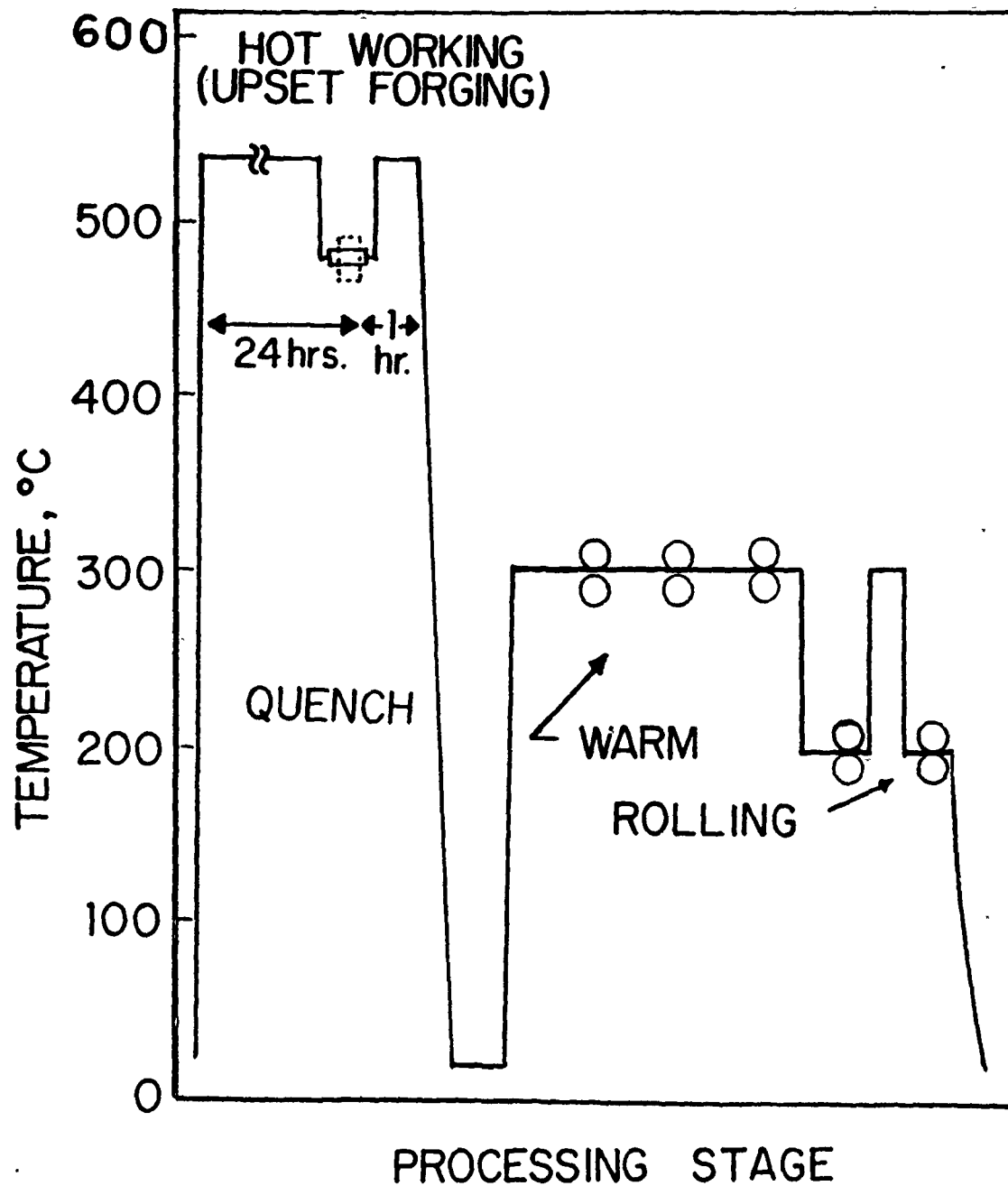


Figure 7. TMP F

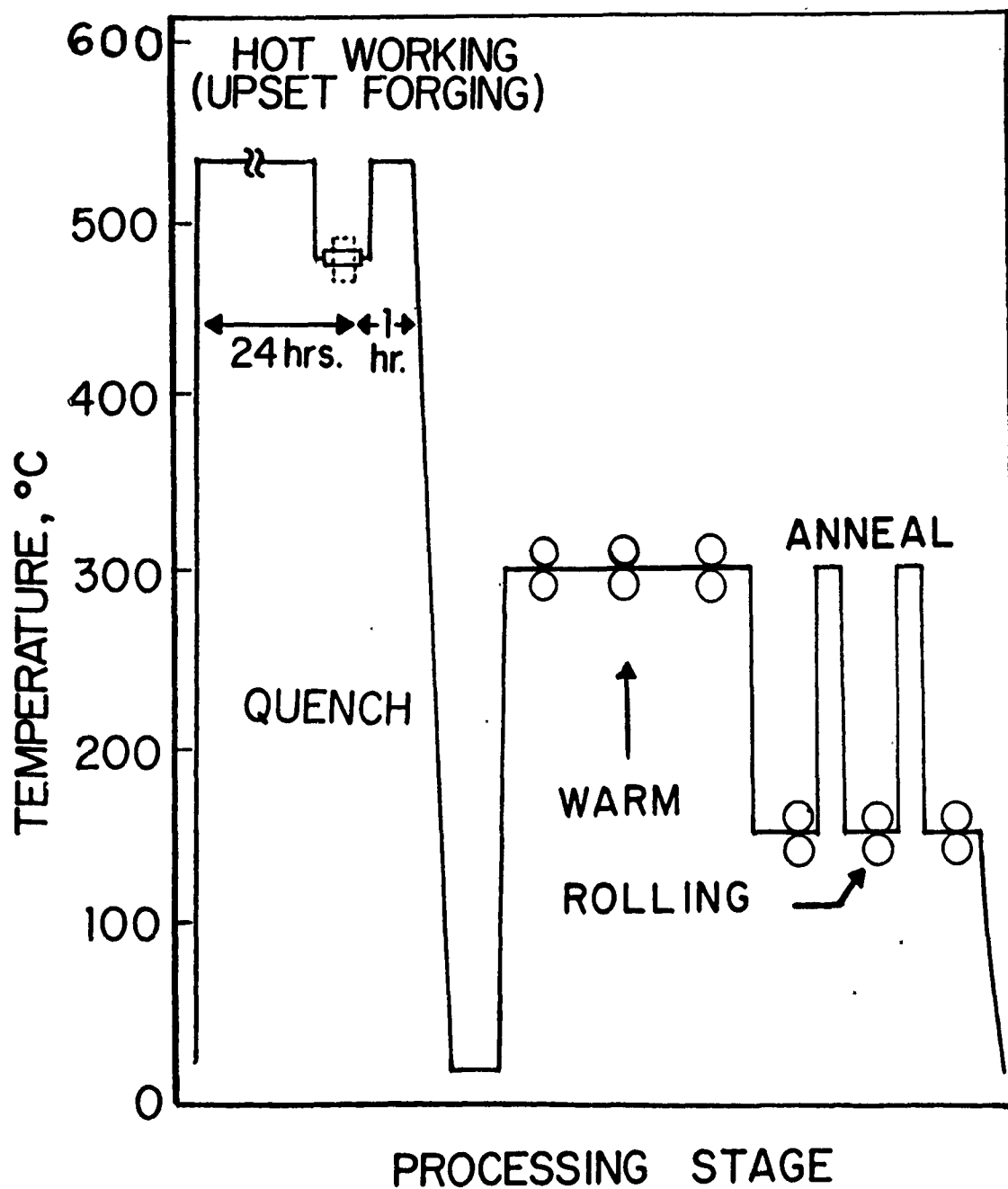


Figure 8. TMP G

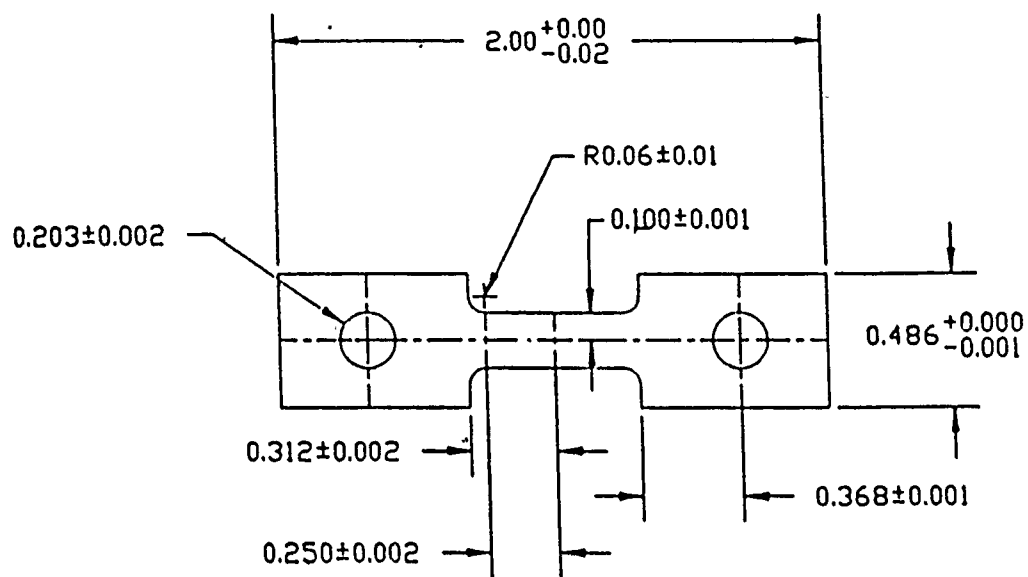


Figure 9. Tensile Test Specimen

IV. RESULTS AND DISCUSSION

This program of research investigated processing of both Al-10Mg-0.1Zr and 2090 alloys. The results of this research regarding each of these alloys will be presented separately.

A. AL-10MG-0.1ZR

In previous research by Gorsuch, three different TMP schedules were employed to process material for study. These TMPs all used the same schedule of rolling reductions at 300°C and differed only in the length of the reheating interval between successive rolling passes. The reheating intervals were 5 minutes, 12.5 minutes and 30 minutes, and were selected based on earlier studies [Refs. 8, 15].

This research incorporated the same rolling schedule into the TMP utilized (TMP A), but with an extension of the 300°C annealing interval time to 60 minutes. The results of tensile testing samples from this rolled material at 300°C are summarized in Figures 10 and 11. The data of this program is compared with that of Gorsuch. Depicted in Figure 10 for the 60 minute anneal material is a peak ductility slightly greater than 400%. Although this is superplastic response, it is significantly less than the ductility achieved by material which had been annealed at 30 minutes between passes, and is also less than the ductility achieved by material annealed by 12.5 minute intervals. The ductility data clearly indicates that as the reheating interval is increased, the ductility increases to a maximum for the 30 minute interval and that upon further reheating, ductility decreases. The flow stress at 10 percent strain data shown in Figure 11 is mutually consistent with the ductility data, and shows the material becoming progressively weaker as the reheating interval is increased from 5 to 30 minutes, but then exhibiting a sharp increase in strength as the interval time is further increased to 60 minutes. It has been proposed that the increase in reheating time from 5 to 30 minutes facilitates continuous recrystallization. For a longer annealing time, formation of an increasingly well defined subgrain structure occurs in the initial rolling/reheating cycles such that the β -phase precipitates on triple junctions and stabilizes the structure. This allows for the introduction of fresh dislocations during the subsequent rolling pass, and with sufficient reheating time these new dislocations merge with the pre-existing boundaries. The result is a progressive, continuous recrystallization reaction. This alloy requires at least 30 minutes of annealing for recovery to allow absorption of further further dislocation in-

roduced by subsequent rolling passes [Ref. 13]. The decrease in ductility of the material resulting from the 60 minute reheating interval is interpreted as being caused by the onset of grain growth resulting from the extension of the recovery time. At a given strain rate, the flow stress increases with the size of the grains as grain growth occurs in a superplastic material. Thus, a balance appears to exist for this alloy involving dislocation generation during straining, recovery, precipitation of the intermetallic β -phase in grain growth.

This supports the concept that by controlling the process, high ductilities can be achieved at relatively low temperatures.

Similar processing studies have been attempted on other aluminum based alloys. Initial attempts to apply this same concept with 2090 alloy have produced a similar trend in ductility and flow stress following the processing at 300°C. However, a much lower ductility is observed, on the order of 100-250 percent. High ductility at low temperature in the absence of cavitation can be attained by control of these parameters. Fine structures were produced and precipitated T_2 particles appeared to stabilize sub-boundaries by forming at triple junctions. However, the boundaries apparently are of insufficient mis-orientation to support superplastic deformation mechanisms [Ref.14].

In the model which was proposed, dislocations are introduced during a rolling pass and then allowed to recover during reheating. The presence of magnesium in the alloy results in substantial strengthening at elevated temperatures and hence requires generation of more dislocations. It may be that the addition of copper and lithium do result in as high a flow stress in the material and thus fewer dislocations are generated. In an effort to raise the dislocation density during processing of the Al-Cu-Li alloy, the rolling temperature was reduced. The annealing temperatures may have to be increased to allow for the recovery of these added dislocations, since annealing at the low temperature utilized for rolling may be too low for recovery to take place. The approach is then to separate the annealing temperatures from that of the deformation.

The first question raised concerns the effect of such a two-temperature process on the Al-10Mg-0.1Zr alloy. Thus, TMP B and TMP C were prepared and used to process Al-10Mg-0.1Zr material. The results are summarized in Figures 12 and 13. In both cases, the results are compared with the 60 minute annealing interval data.

It is evident from Figure 12 that the material processed with the longer annealing interval of 30 minutes at 300°C (after rolling at 250°C) (TMP B) has the effect of shifting down the strain rate where the peak ductility occurs. The material processed with the shorter annealing interval of 15 minutes (TMP C) exhibits a superplastic response very

similar to that of material processed with a 12.5 minute interval in Gorsuch (Figure 10). This suggests that no further refinement has been obtained by reducing the rolling temperature. The increased dislocation density developed may drive up the rate of grain growth faster than the development of boundary misorientation. The flow stress data represented in Figure 13 are consistent with this as well.

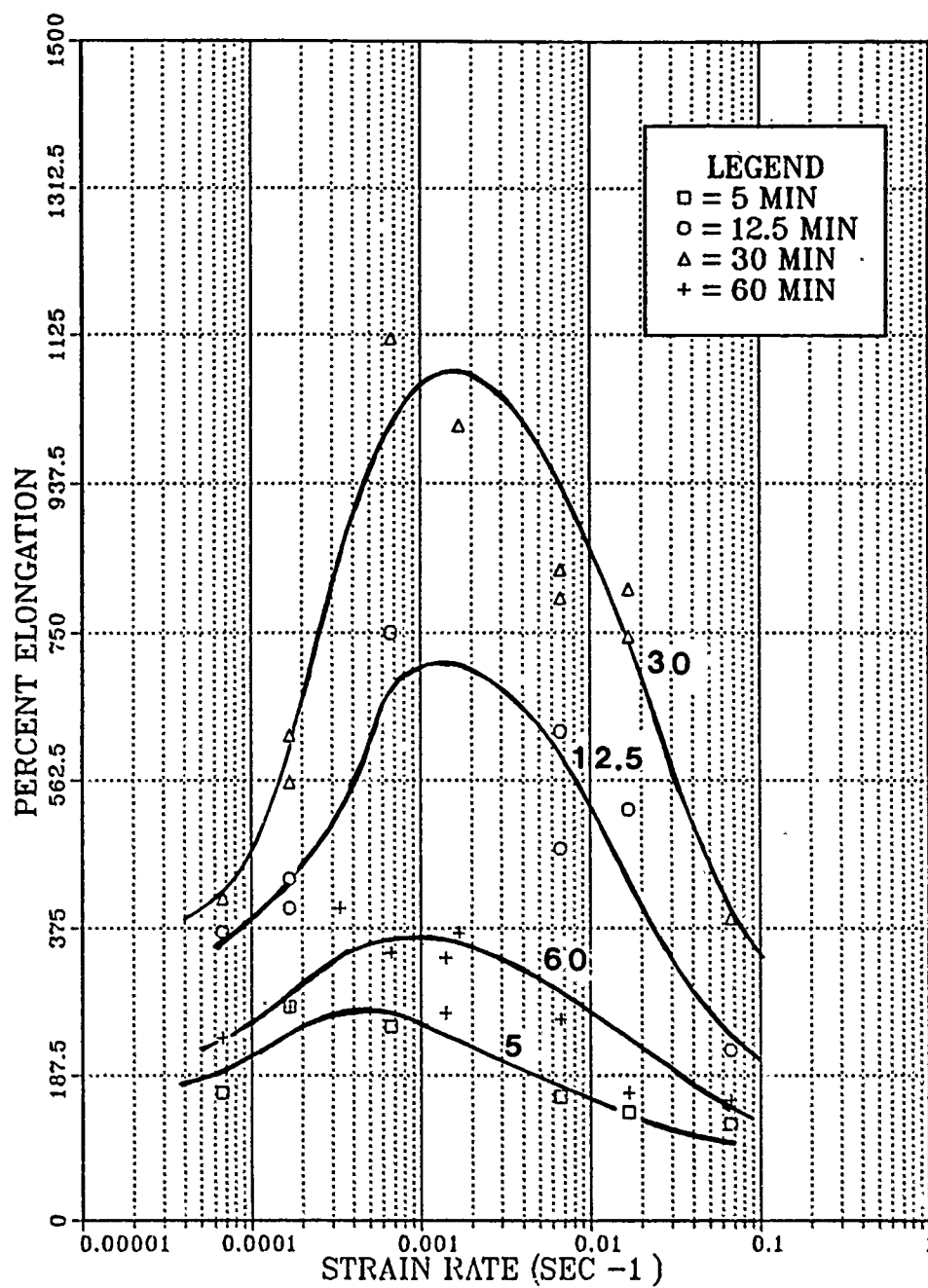


Figure 10. Al-10Mg Ductility vs. Strain Rate

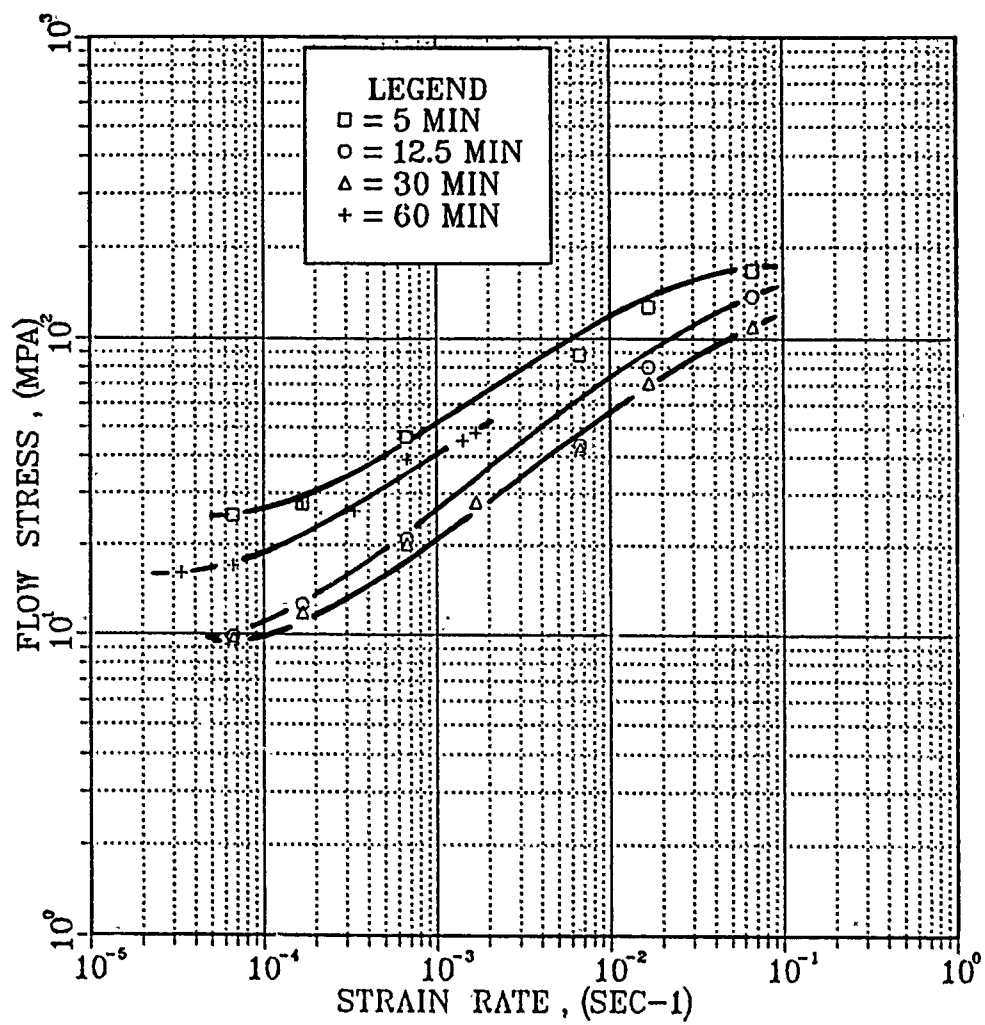


Figure 11. Al-10Mg Flow Stress vs. Strain Rate

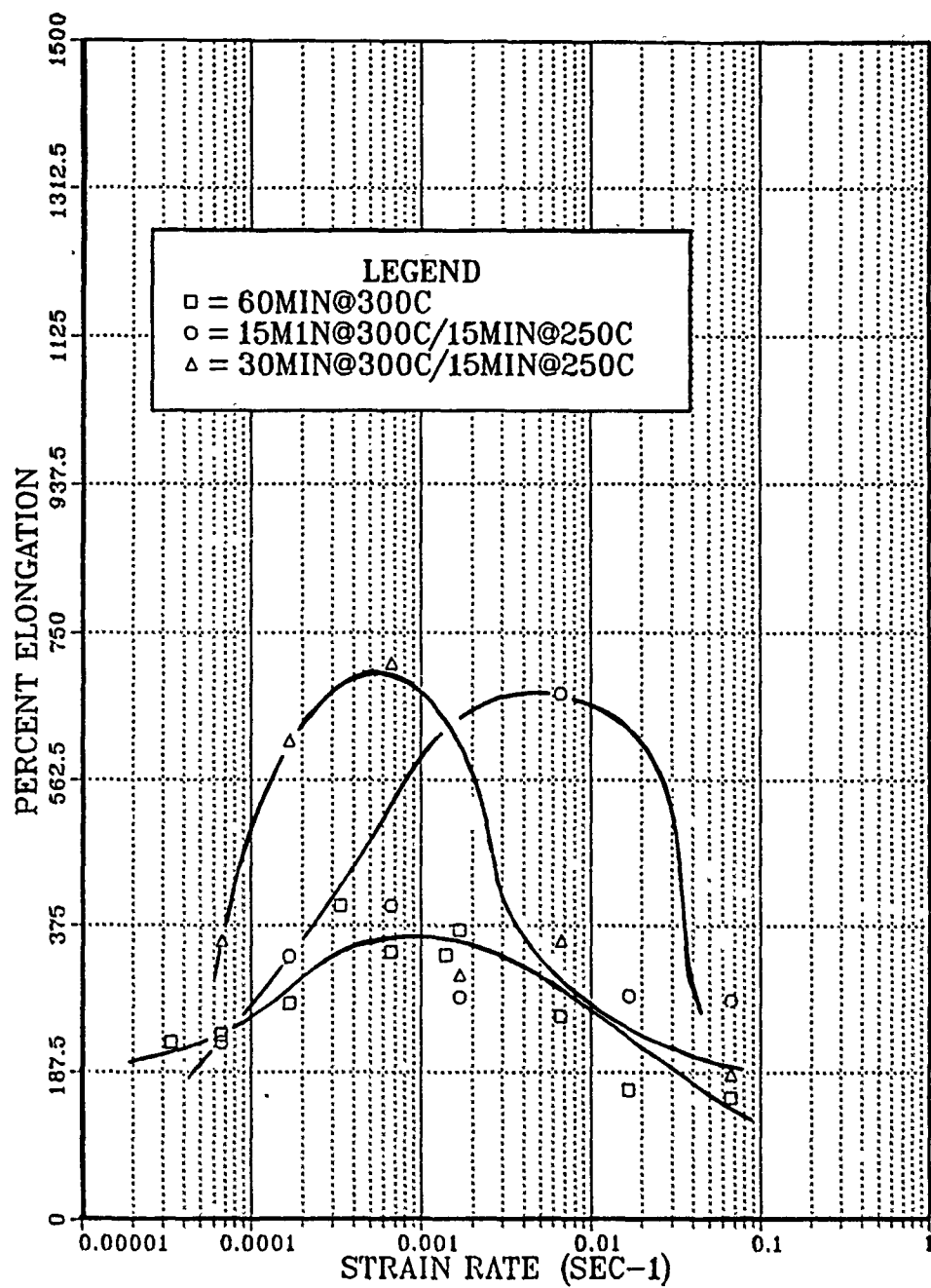


Figure 12. Al-10Mg Ductility vs. Strain Rate

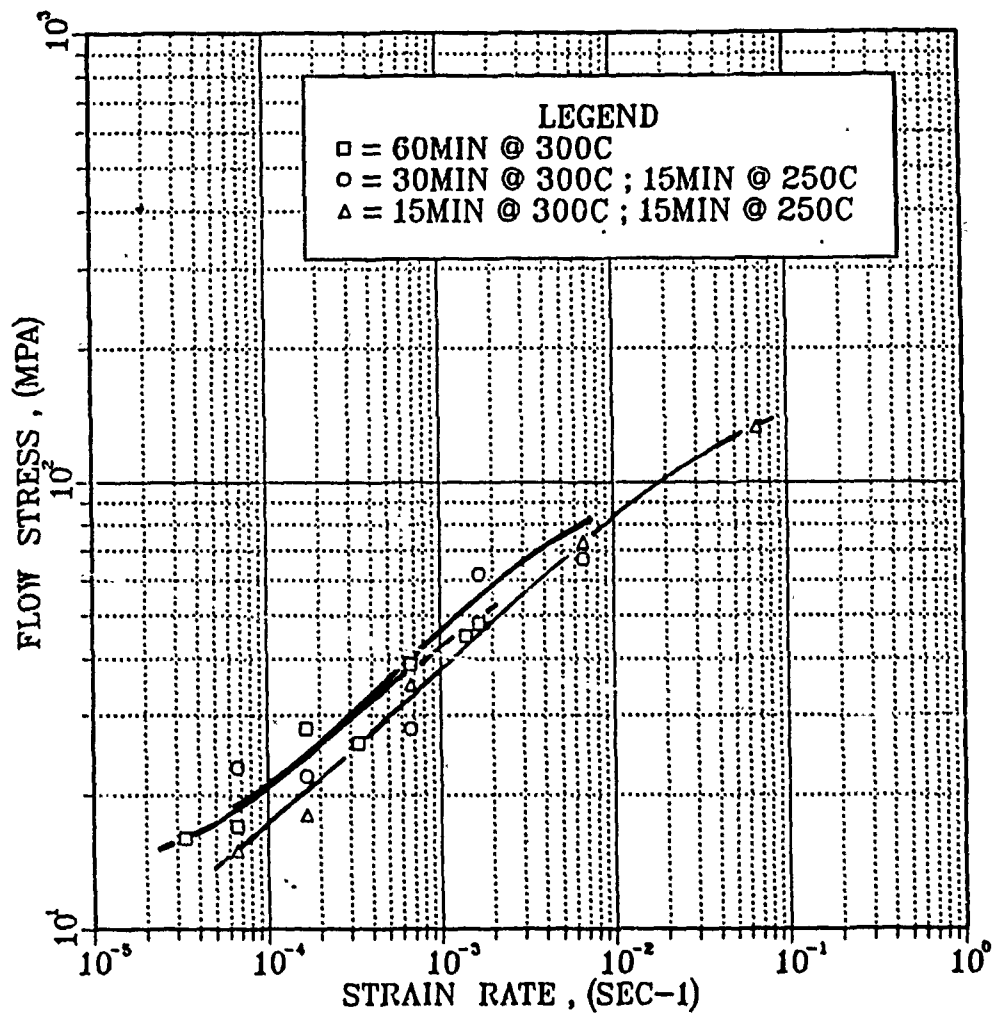


Figure 13. Al-10Mg Flow Stress vs. Strain

B. 2090

This research also investigated the effects of processing variations on this alloy. Initial efforts examined the effect on ductility when the annealing time was extended to 60 minutes between rolling passes for rolling conducted at 300°C. The results of this experiment are summarized in Figures 14 and 15. The ductility data in Figure 14 display a characteristic peak in ductility, similar to that obtained with Al-10Mg-0.1Zr. In the case with 2090 however, the peak ductility is obtained at a substantially higher temperature, 425°C, as compared with 300°C for Al-10Mg-0.1Zr. It is noteworthy also that peak ductility in 2090 occurred approximately 60°C below the T_2 phase solvus temperature. The peak ductility of Al-10Mg-0.1Zr occurred approximately 60°C below the β -phase solvus temperature.

The flow stress vs. temperature curves in Figure 15 show a characteristic weakening with increased temperature, and then a sharp increase in strength between 425°C and 450°C. This is attributed to grain growth as the material approaches the T_2 phase solvus temperature and the stabilizing effect of the T_2 is lost. Similar effects have been reported for the Al-10Mg-0.1Zr alloy [Ref.15]. An important observation to make for the 2090 alloy is that ductility is increased when the annealing time is increased from 5 minutes to 30 minutes and there on to 60 minutes. This suggests that 2090 recovers more slowly at 300°C than the Al-10Mg-0.1Zr alloy.

Next, the annealing temperature was maintained at 300°C while the rolling temperature was lowered. The annealing interval prior to each rolling pass was 15 minutes each. These parameters were chosen to investigate the effect on ductility of rolling at reduced temperatures with only a short annealing interval.

The first of this dual temperature series employed a 250°C rolling temperature. The results are summarized in Figures 16 and 17. Peak ductility was again achieved at 425°C as in the 60-minute anneal series. This ductility is greater than that achieved in previous research when the alloy was rolled at 300°C [Ref. 13]. The flow stress results depict the expected loss of strength with the increase of temperature, followed as well by the characteristic increase (at the lower strain rates) in strength once the alloy passes 425°C. These results suggest that the schedule involving the lower rolling temperature has a beneficial effect upon refinement of the grain structure.

The next experiment is one wherein the rolling temperature was reduced to 200°C. The results are shown in Figures 18 and 19. The ductility achieved with this process is lower than that of the previous process where the rolling temperature was 50°C higher. The flow stress and ductility data supports the contention that some microstructural

refinement has taken place. The final experiment with separate annealing and deformation temperatures is that involving a rolling temperature of 150°C. Figures 20 and 21 contain the results of this work. It can be seen that ductility has now increased above that of the 200°C rolling experiment, although not to the extent obtained with the 250°C rolling. The flow stress data is mutually consistent with that of the ductility results, and suggests again that the grain refinement required for superplastic elongation has occurred but not to the same extent as with the Al-10Mg-0.1Zr alloy.

All three of the reduced-rolling-temperature experiments produced superplastic tensile elongations. All of the peak ductilities occurred at a tensile test temperature of 425°C, and a strain rate of $6.67 \times 10^{-3} \text{ sec}^{-1}$. This increased ductility obtained as a result of lowering the rolling temperatures indicates that the dislocation density has been increased but that recovery has not provided a refined structure of sufficiently misoriented boundaries for a fully superplastic response.

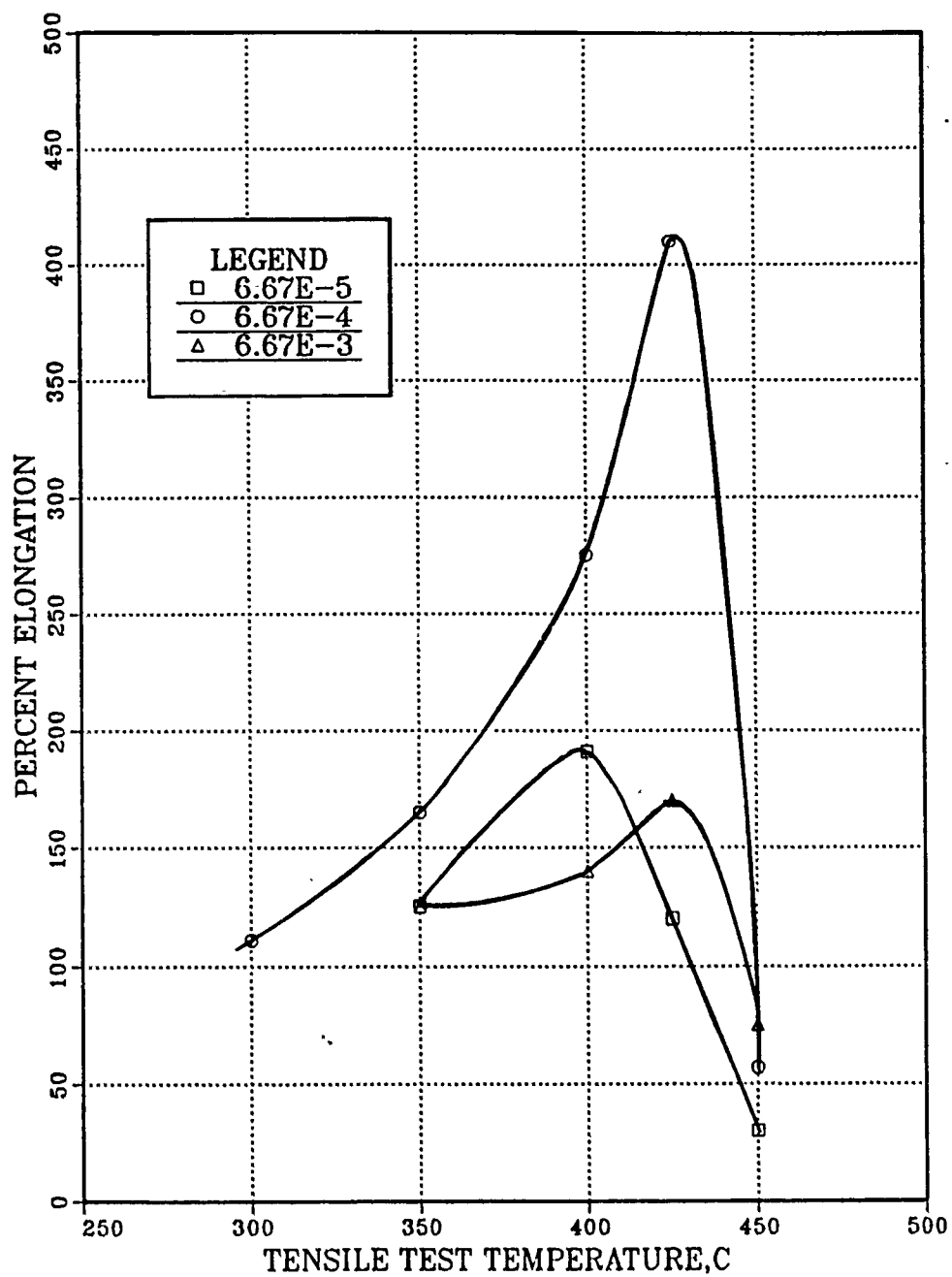


Figure 14. 2090 Ductility vs. Tensile Test Temperature: 60 minute anneal at 300°C

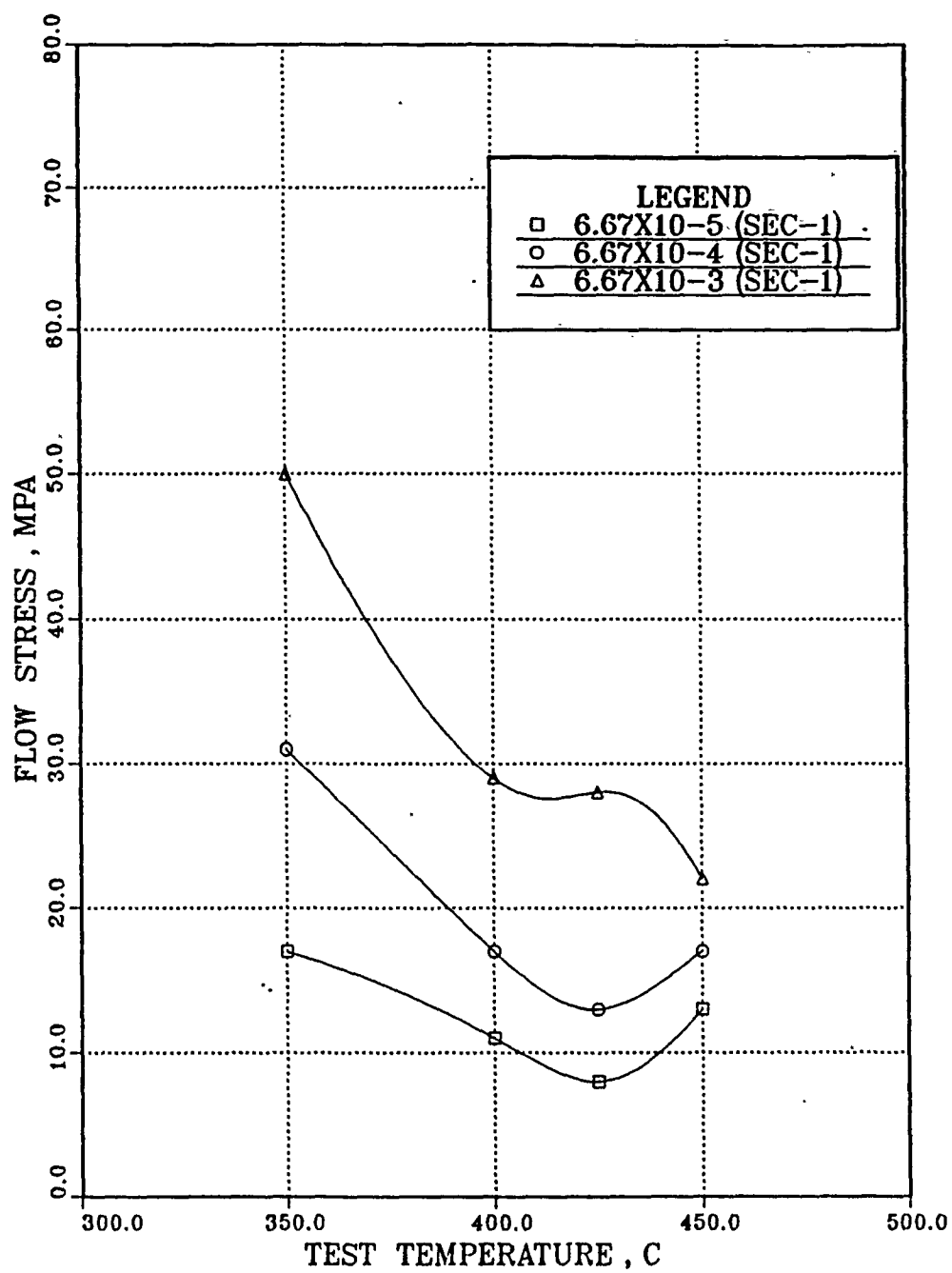


Figure 15. 2090 Flow Stress vs. Tensile Test Temperature: 60 minute anneal at 300°C

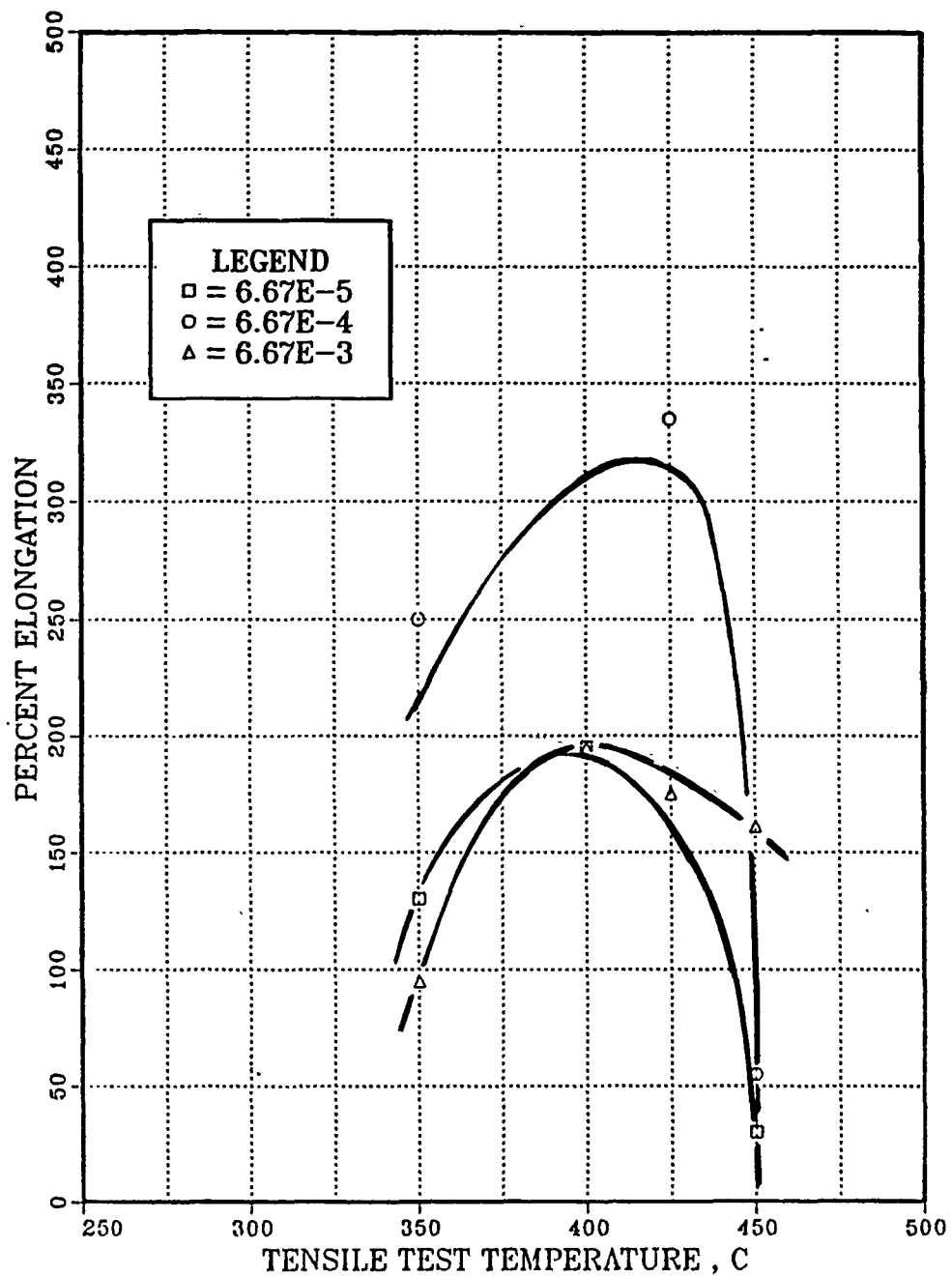


Figure 16. 2090 Ductility vs. Tensile Test Temperature: 15 minutes at 300°C, 15 minutes at 250°C

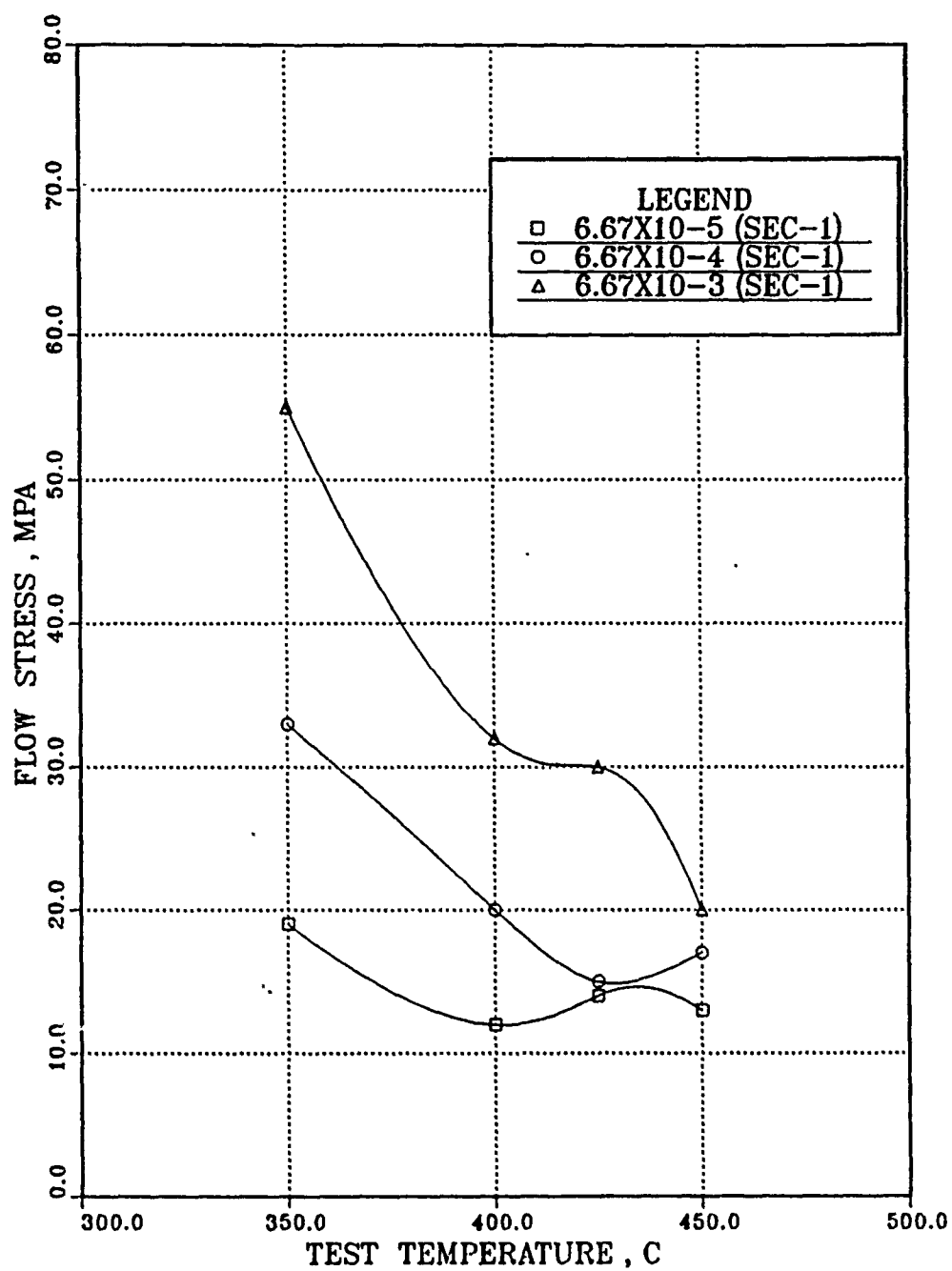


Figure 17. 2090 Flow Stress vs. Tensile Test Temperature: 15 minutes at 300°C,
15 minutes at 250°C

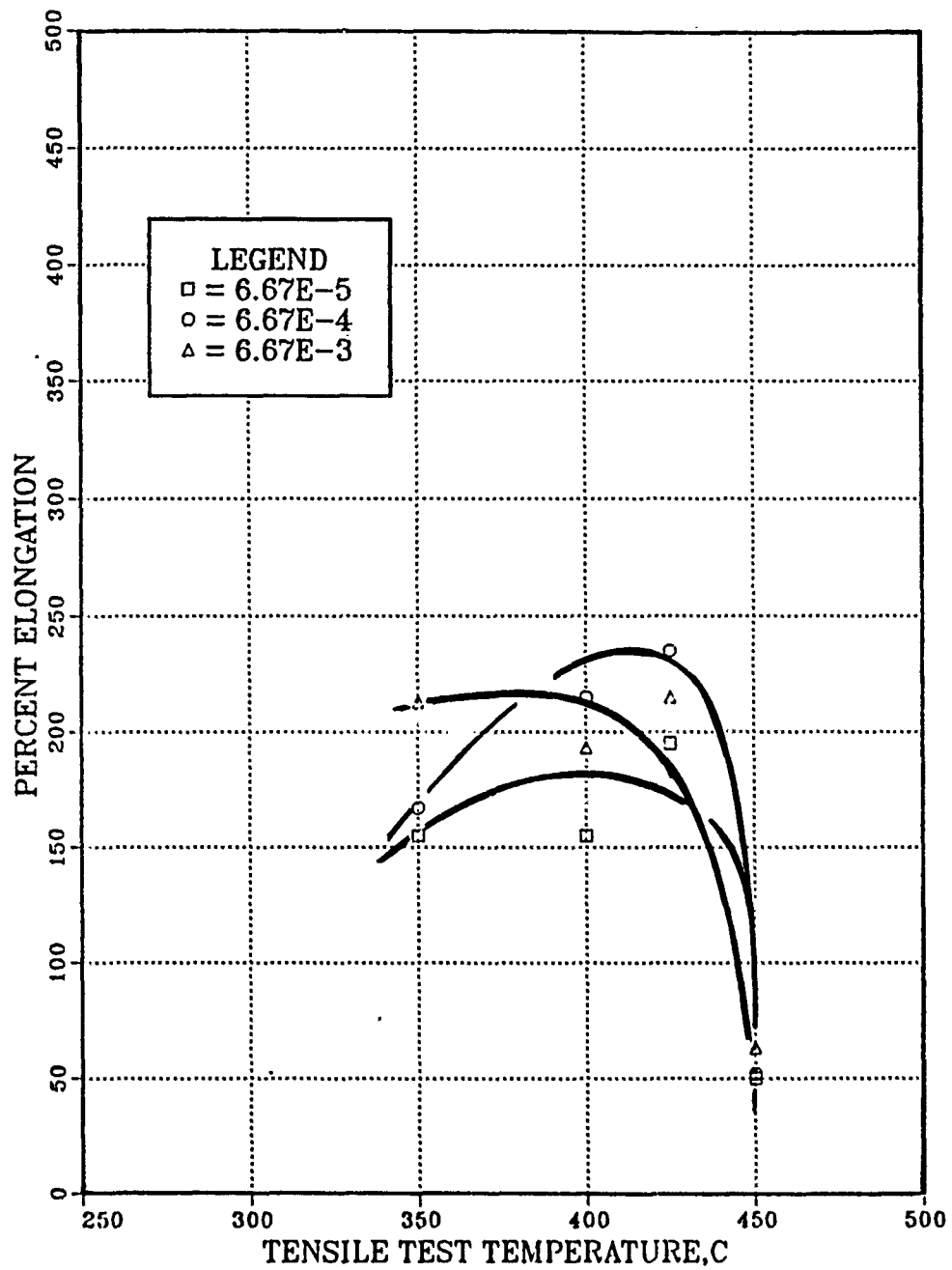


Figure 18. 2090 Ductility vs. Tensile Test Temperature: 15 minutes at 300°C, 15 minutes at 200°C

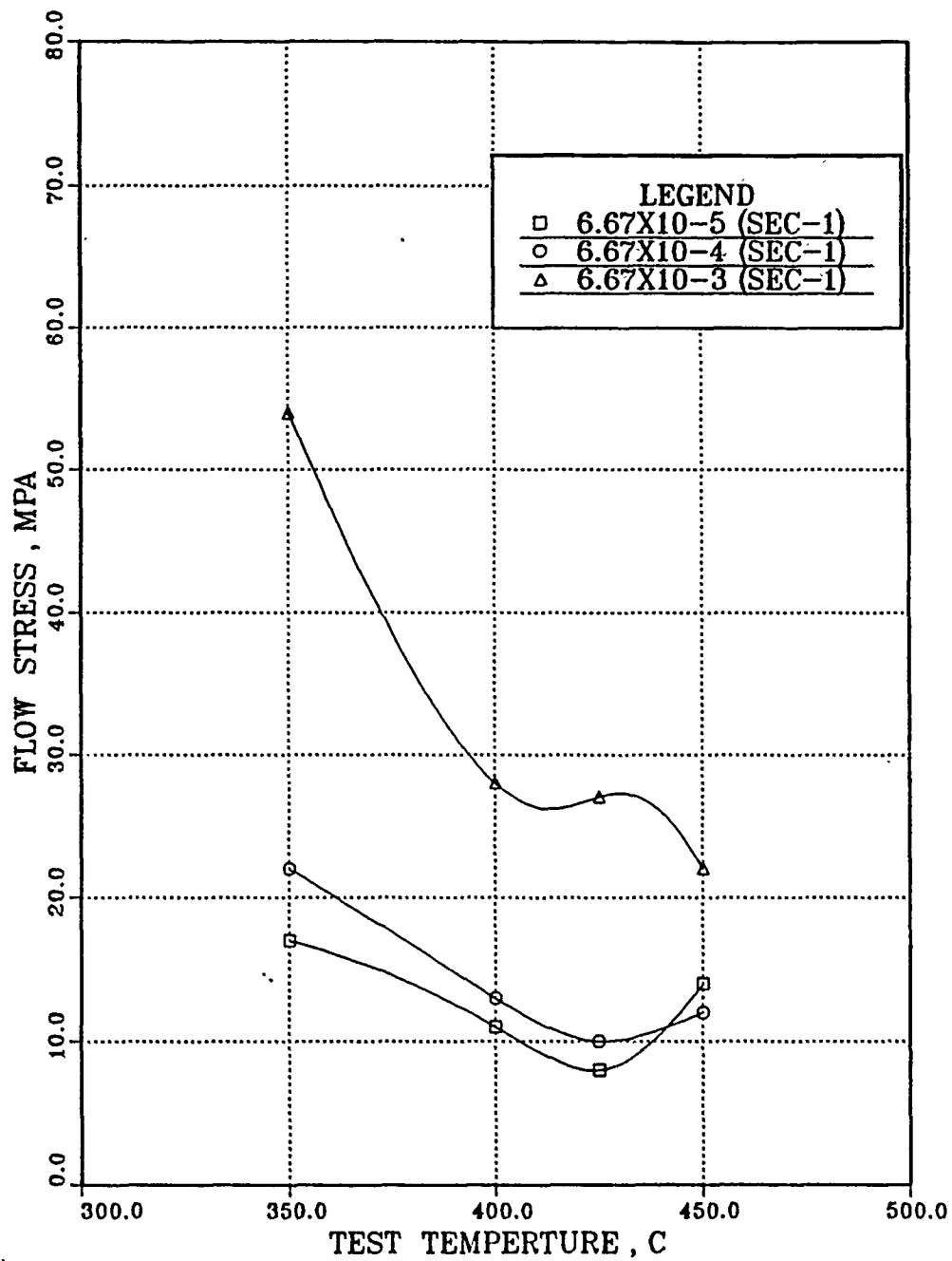


Figure 19. 2090 Flow Stress vs. Tensile Test Temperature: 15 minutes at 300°C, 15 minutes at 200°C

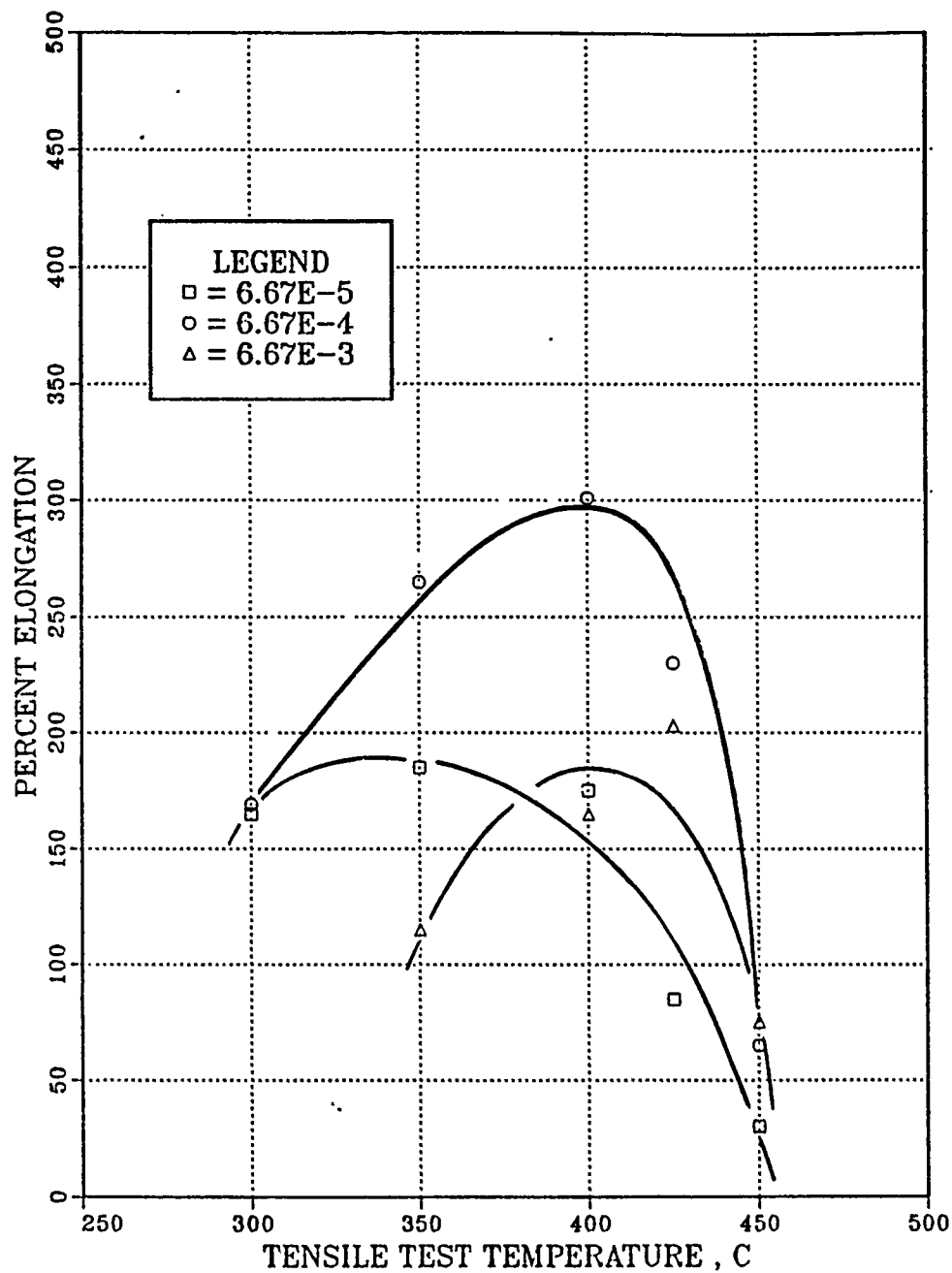


Figure 20. 2090 Ductility vs. Tensile Test Temperature: 15 minutes at 300°C, 15 minutes at 150°C

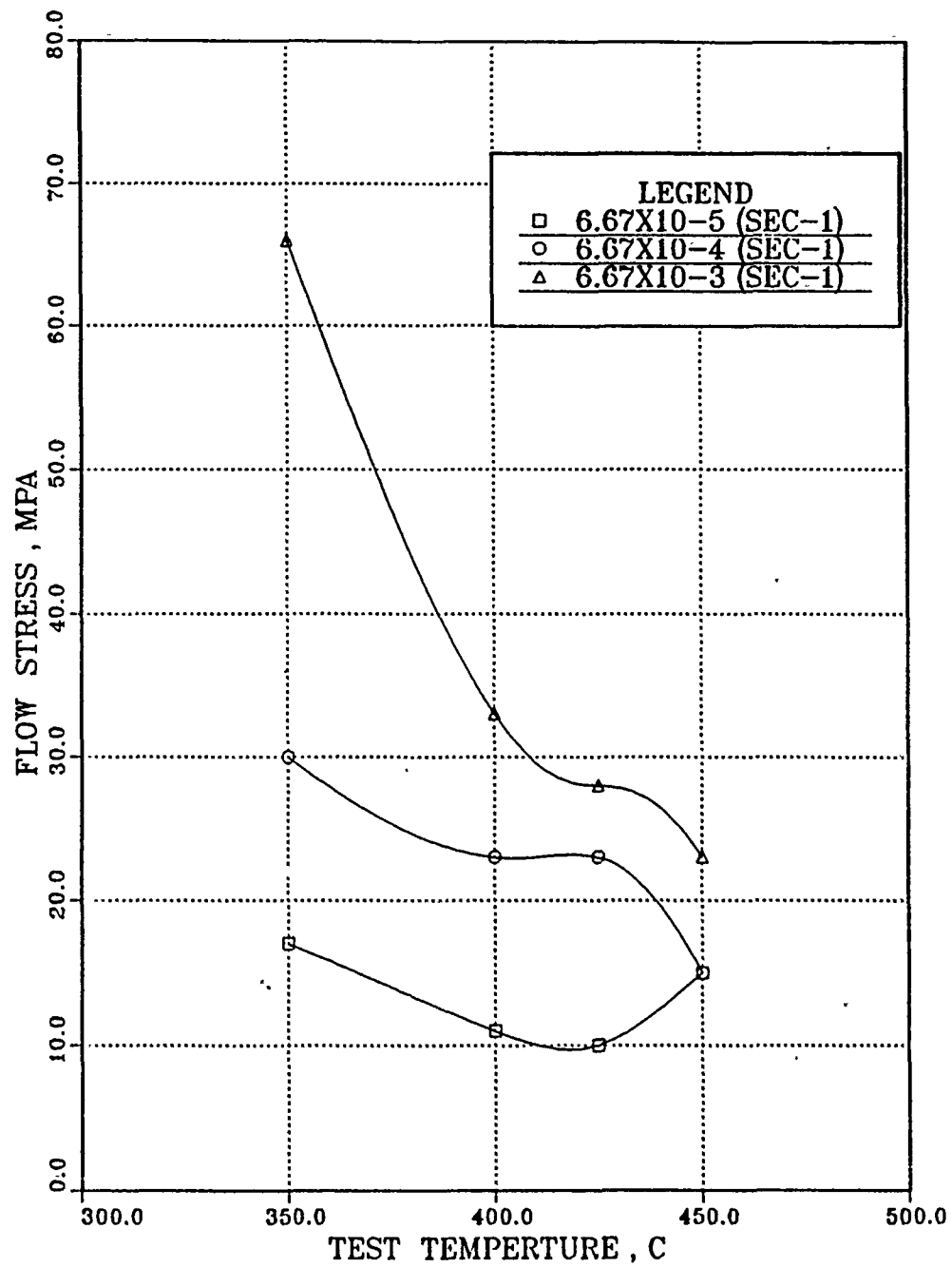


Figure 21. 2090 Flow Stress vs. Tensile Test Temperature: 15 minutes at 300°C, 15 minutes at 150°C

C. SUMMARY

This thesis has investigated the effects on the superplastic behavior of two aluminum alloys produced by variation of the thermomechanical process employed. The alloys studied were Al-10Mg-0.1Zr and 2090. With the Al-10Mg-0.1Zr, this research continued previous work by the extension of annealing time between rolling passes. The objective here was to determine whether an optimum balance between deformation and recovery exists. By combining the results of Gorsuch with those of this research, it is clear that the optimum annealing time is 30 minutes for this alloy when processed with the utilized rolling schedule at 300°C. The total strain induced during this process is 2.5. Upon further increase of annealing time, the microstructure is believed to experience grain growth, resulting in a coarser grain structure and reduced ductility.

In the course of research on processing of the 2090 alloy, a dual-temperature deformation and recovery method was proposed. The logical question that arose initially concerned the effect on ductility if this process were applied to the Al-10Mg-0.1Zr alloy. To address that question, two dual-temperature processes were conducted. Both of the processes employed annealing at 300°C and rolling at 250°C. The difference between the two was the time of annealing between rolling passes. The first, (TMP B) employed a 30 minute recovery time whereas the second, (TMP C) utilized a 15 minute interval. Although both processes produced superplastic response, the ductility obtained was less than that associated with using the optimum processing procedure, (TMP A). The reason for this may be that the dislocation density produced as a result of the cooler rolling is raised beyond the point at which the recovery times are now too low, or grain growth is occurring.

Research with the 2090 alloy began with an extension of the annealing interval to 60 minutes (TMP D). This process produced the greatest elongation to date with this alloy in the research conducted at NPS. By extending the annealing interval after the rolling passes, the microstructure produced has been stabilized by the precipitation of the T_2 phase. A fine, stable structure is required for superplasticity. These results suggest that by extending the annealing time even further, enhanced superplasticity may result. By applying dual-temperature deformation and recovery cycles to the 2090, increased ductility also resulted. This likely is a result of an increased dislocation density introduced during the lower temperature rolling. This increase in ductility resulted while employing recovery times of only 15 minutes. These results suggest that even further enhancement may result by combining extended recovery time and reduced rolling temperatures. All TMP ductility results are presented in Tables 4-10 below.

Table 4. DUCTILITY AND FLOW STRESS OF TMP A (10 MG)

Strain Rate (sec ⁻¹)	Ductility (percent)	Flow Stress at 10% strain (MPa)
3.34×10^{-5}	225	16
6.67×10^{-5}	235	17
1.67×10^{-4}	275	28
3.34×10^{-4}	400	26
6.67×10^{-4}	341	39
1.40×10^{-3}	337	45
1.67×10^{-3}	369	48

Table 5. DUCTILITY AND FLOW STRESS OF TMP B (10 MG)

Strain Rate (sec ⁻¹)	Ductility (percent)	Flow Stress at 10% strain (MPa)
6.67×10^{-5}	355	15
1.67×10^{-4}	613	18
6.67×10^{-4}	711	35
6.67×10^{-3}	355	73
6.67×10^{-2}	185	133

Table 6. DUCTILITY AND FLOW STRESS OF TMP C (10 MG)

Strain Rate (sec ⁻¹)	Ductility (percent)	Flow Stress at 10% strain (MPa)
6.67×10^{-5}	225	23
1.67×10^{-4}	335	22
6.67×10^{-4}	400	28
1.67×10^{-3}	283	62
6.67×10^{-3}	672	67

Table 7. DUCTILITY AND FLOW STRESS OF TMP D (2090)

Strain Rate	6.67×10^{-5}		6.67×10^{-4}		6.67×10^{-3}	
Tensile Test Temp.	ductility (pct)	flow stress (MPa)	ductility (pct)	flow stress (MPa)	ductility (pct)	flow stress (MPa)
350°C	125	17	165	31	130	50
400°C	191	11	275	17	140	29
425°C	120	8	410	13	170	28
450°C	30	13	65	17	75	22

Table 8. DUCTILITY AND FLOW STRESS OF TMP E (2090)

Strain Rate	6.67×10^{-5}		6.67×10^{-4}		6.67×10^{-3}	
Tensile Test Temp.	ductility (pct)	flow stress (MPa)	ductility (pct)	flow stress (MPa)	ductility (pct)	flow stress (MPa)
350°C	130	19	250	33	95	55
400°C	195	12	195	20	195	32
425°C	161	14	335	15	175	30
450°C	30	13	55	17	35	20

Table 9. DUCTILITY AND FLOW STRESS OF TMP F (2090)

Strain Rate	6.67×10^{-5}		6.67×10^{-4}		6.67×10^{-3}	
Tensile Test Temp.	ductility (pct)	flow stress (MPa)	ductility (pct)	flow stress (MPa)	ductility (pct)	flow stress (MPa)
350°C	155	17	167	30	213	54
400°C	155	11	215	13	193	28
425°C	195	8	235	10	215	27
450°C	50	14	52	12	63	22

Table 10. DUCTILITY AND FLOW STRESS OF TMP G (2090)

Strain Rate	6.67×10^{-5}		6.67×10^{-4}		6.67×10^{-3}	
Tensile Test Temp.	ductility (pct)	flow stress (MPa)	ductility (pct)	flow stress (MPa)	ductility (pct)	flow stress (MPa)
350°C	185	17	265	30	115	66
400°C	175	11	301	23	165	33
425°C	85	10	230	15	203	28
450°C	30	15	65	17	75	23

V. CONCLUSIONS

1. In Al-10Mg-0.1Zr, extension of annealing time to 60 minutes between rolling passes decreases ductility significantly. thus, the 30 minute annealing interval appears to be the optimum for this alloy undergoing this thermomechanical process.
2. Variation of the rolling temperature from that of the testing and annealing temperature did not enhance ductility in Al-10Mg-0.1Zr.
3. With 2090, the extension of the annealing time to 60 minutes produced and enhanced ductility compared with past research.

VI. RECOMMENDATIONS

1. Investigate the use of longer annealing times between rolling passes with the 2090 in conjunction with lowering the rolling temperature in the two temperature annealing-rolling schedule in order to determine an optimum combination of time and temperature to produce maximum tensile elongation.
2. Investigate Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) to determine the effects on the microstructure by the extended annealing times.

APPENDIX A. ENGINEERING STRESS-STRAIN CURVES

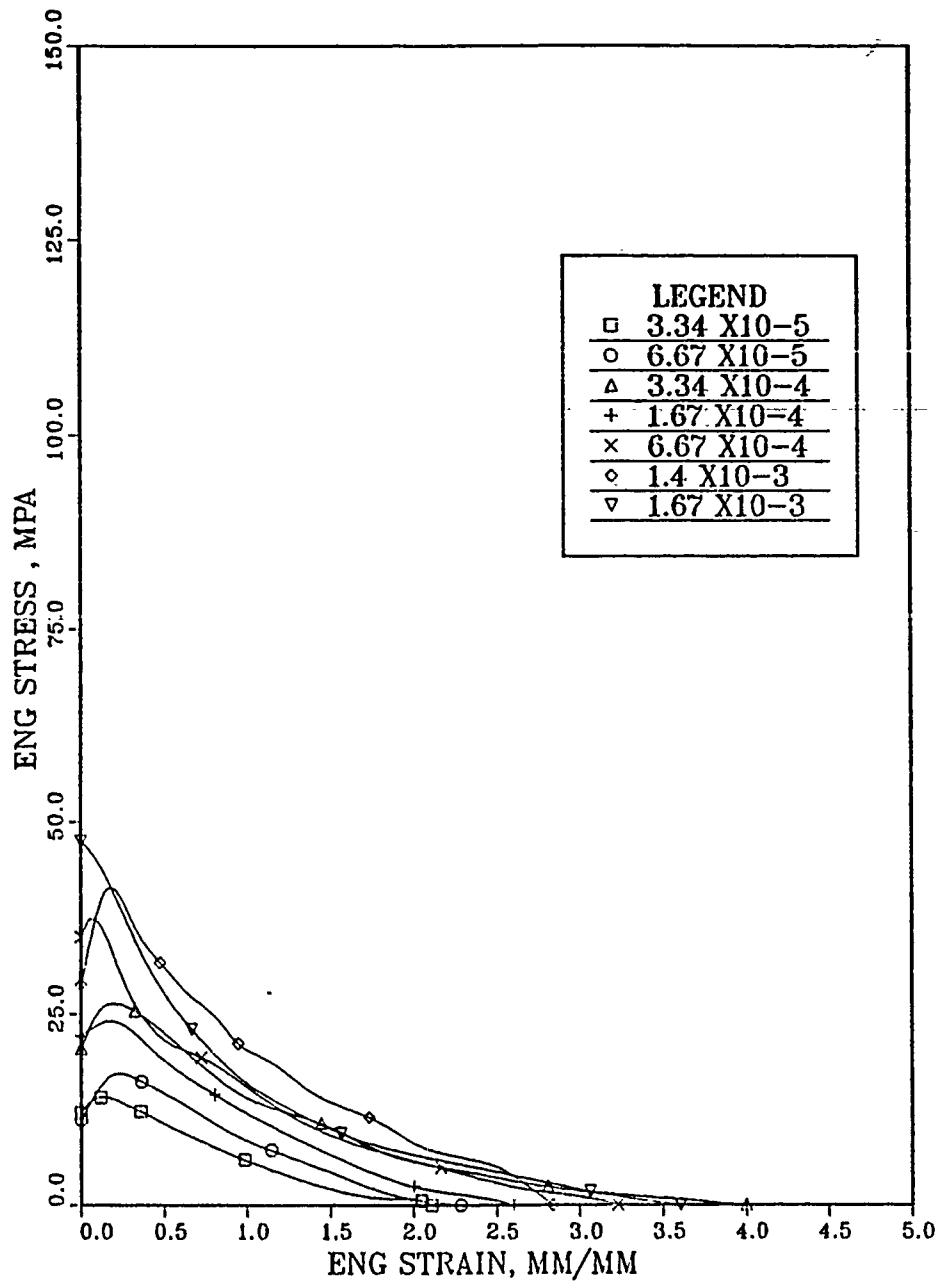


Figure 22. TMP A Al-10Mg-0.1Zr 60min @ 300°C

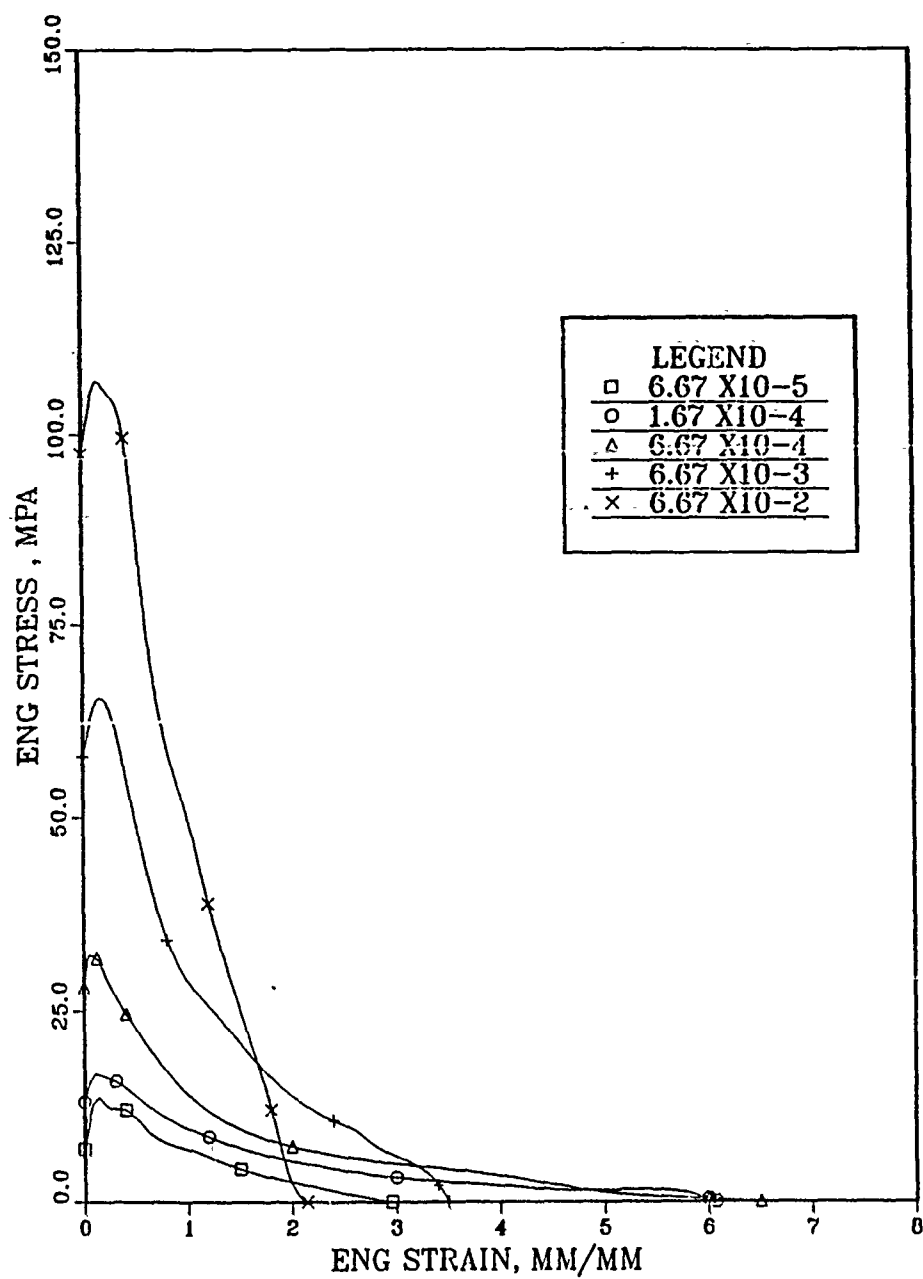


Figure 23. TMP B Al-10Mg-0.1Zr 30min @ 300°C / 15min @ 250°C

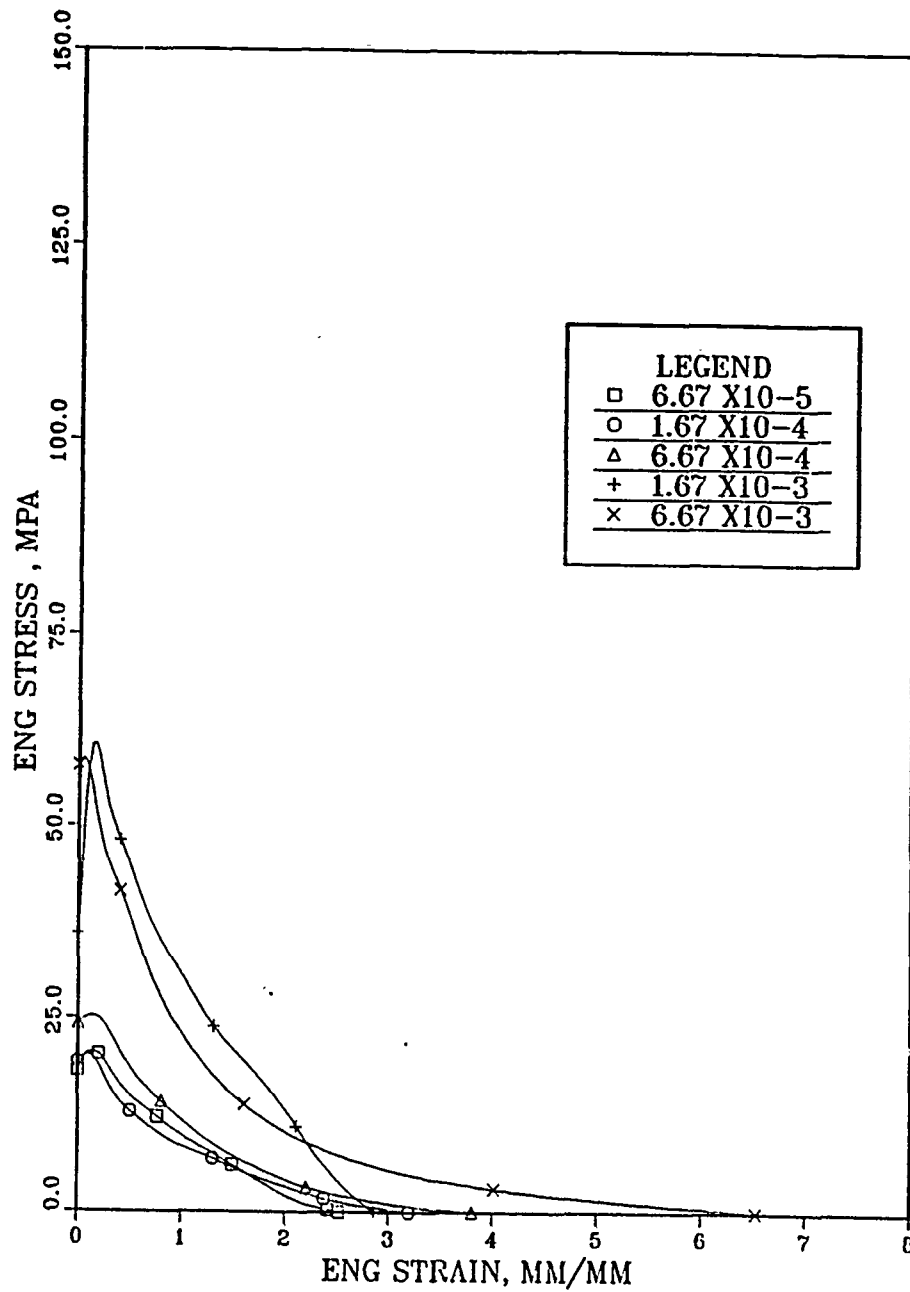


Figure 24. TMP C Al-10Mg-0.1Zr 15min @ 300°C / 15min @ 250°C

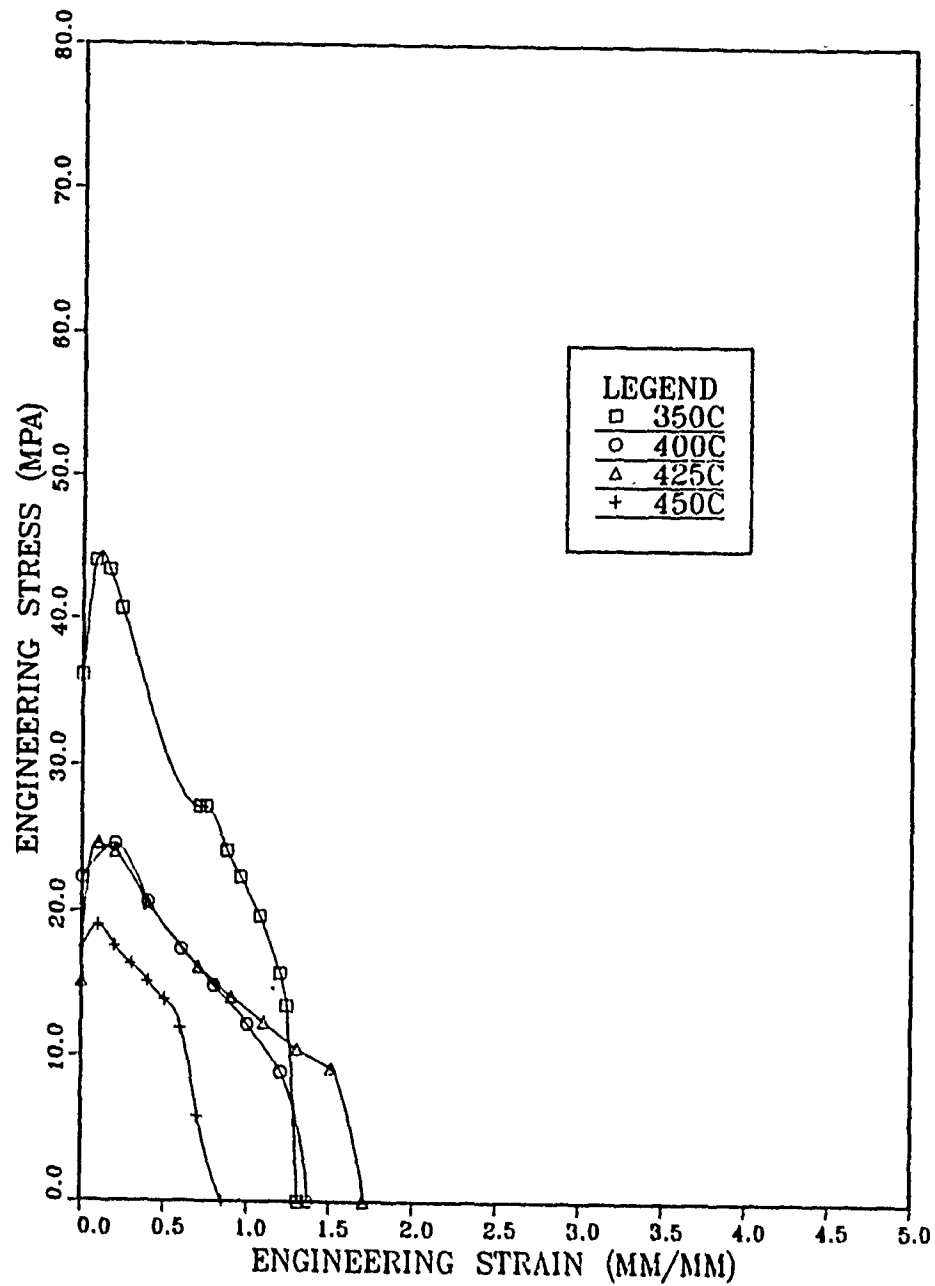


Figure 25. TMP D 2090 60min @ 300°C: Tested at $6.67 \times 10^{-5} \text{ sec}^{-1}$ strain rate

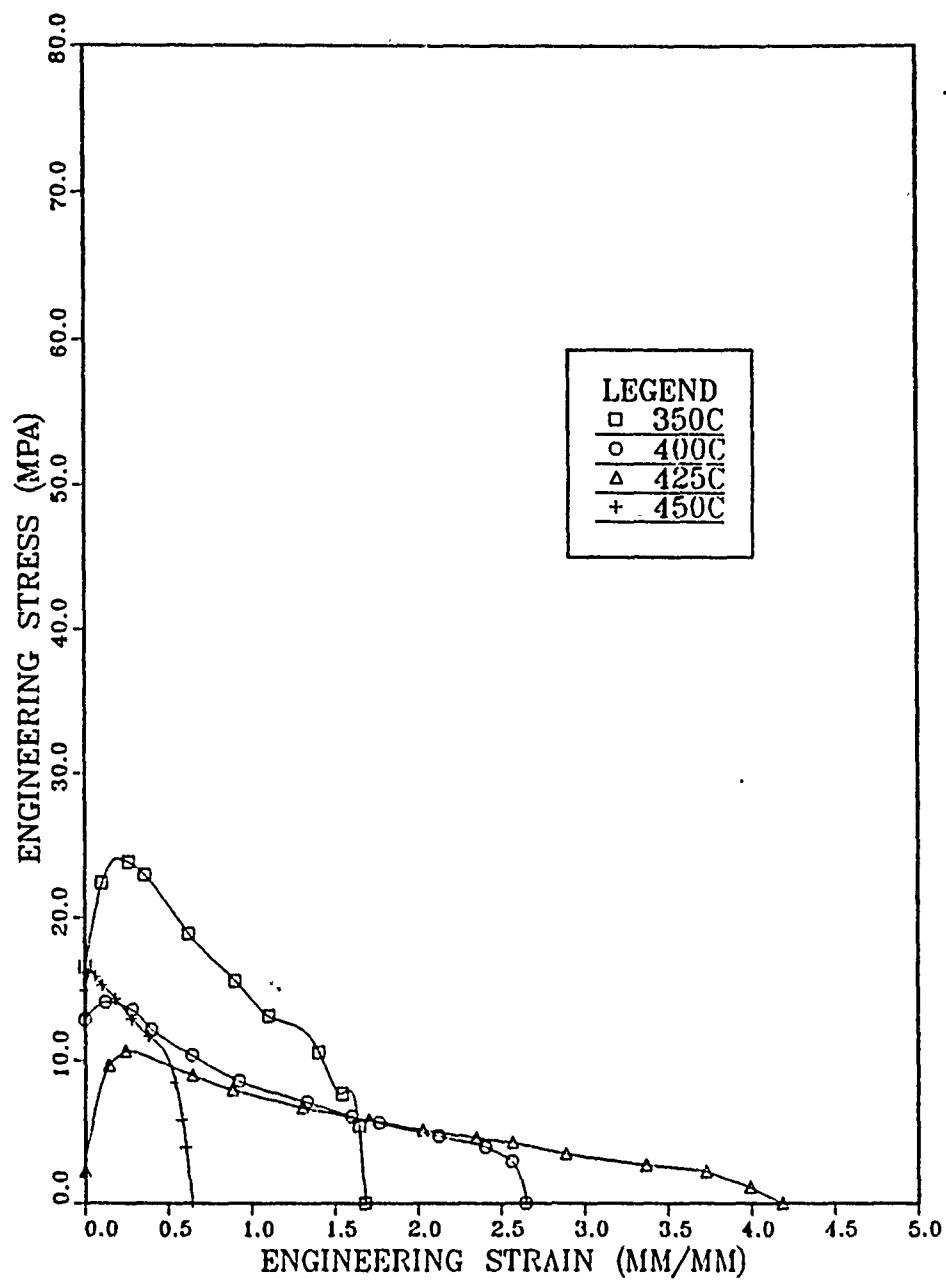


Figure 26. TMP D 2090 60min @ 300°C: Tested at $6.67 \times 10^{-4} \text{ sec}^{-1}$ strain rate

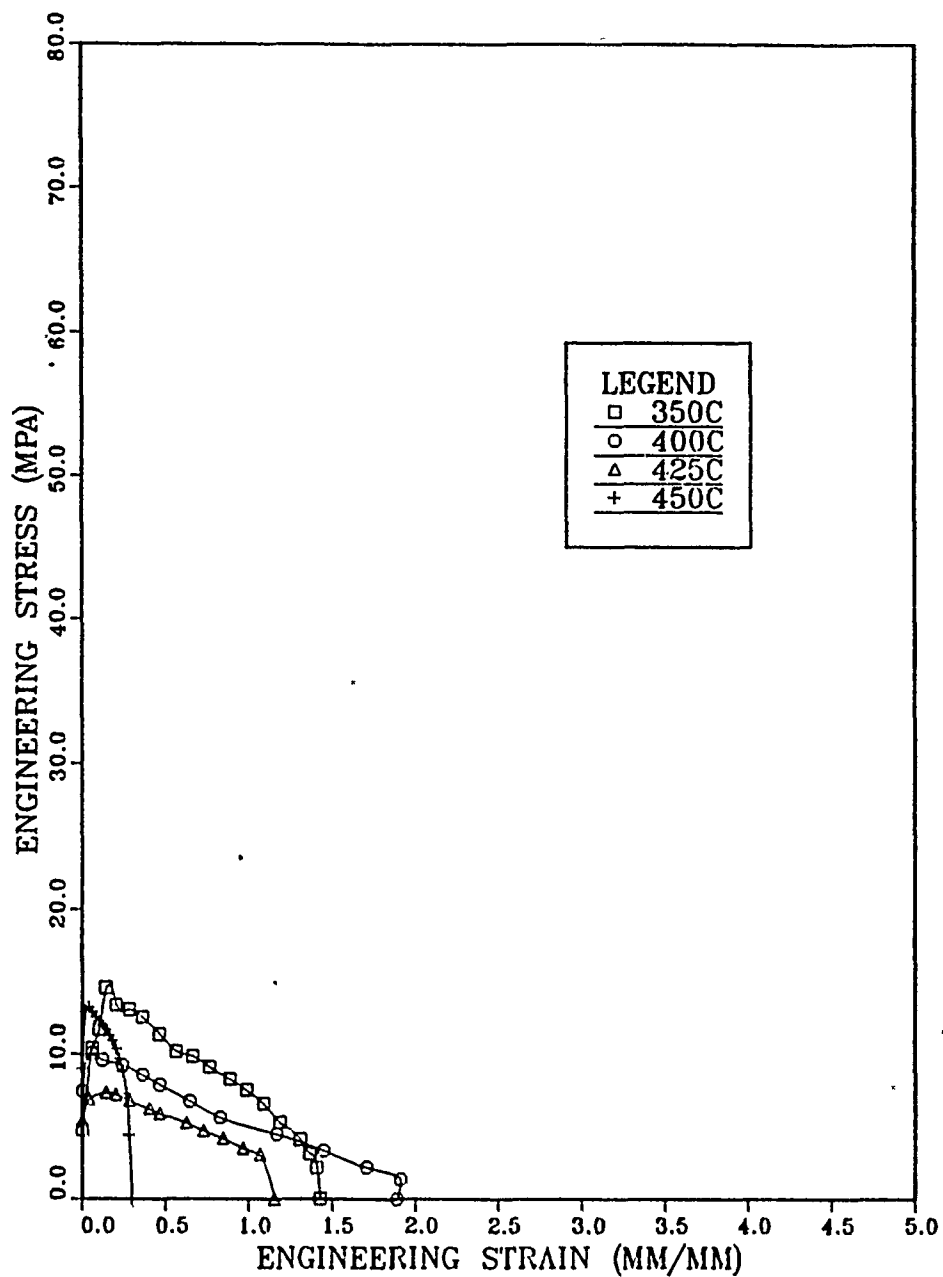


Figure 27. TMP D 60min @ 300°C: Tested at $6.67 \times 10^{-3} \text{ sec}^{-1}$ strain rate

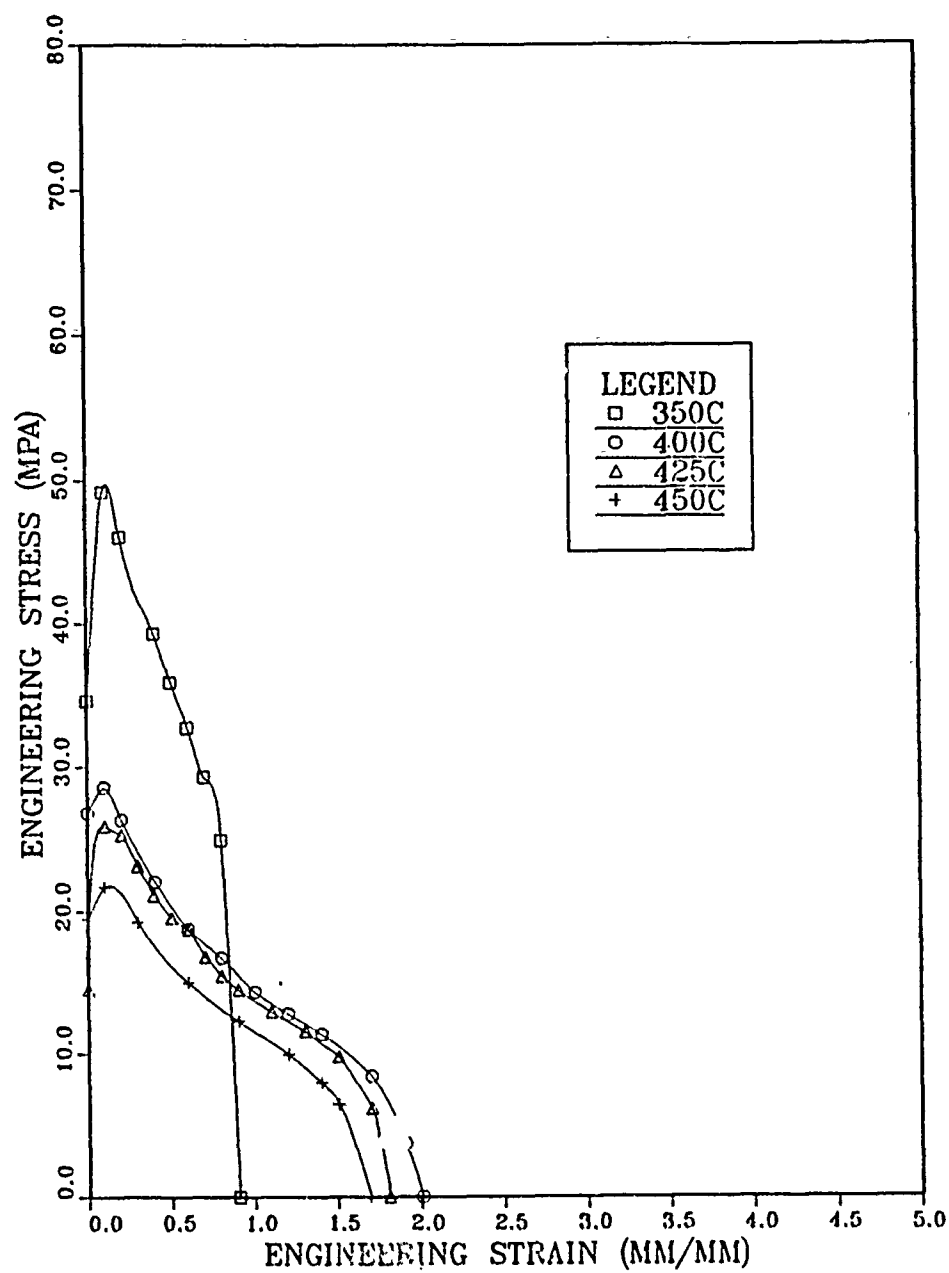


Figure 28. TMP E 2090 15min @ 300°C / 15min @ 250°C: Tested at $6.67 \times 10^{-2} \text{ sec}^{-1}$ strain rate

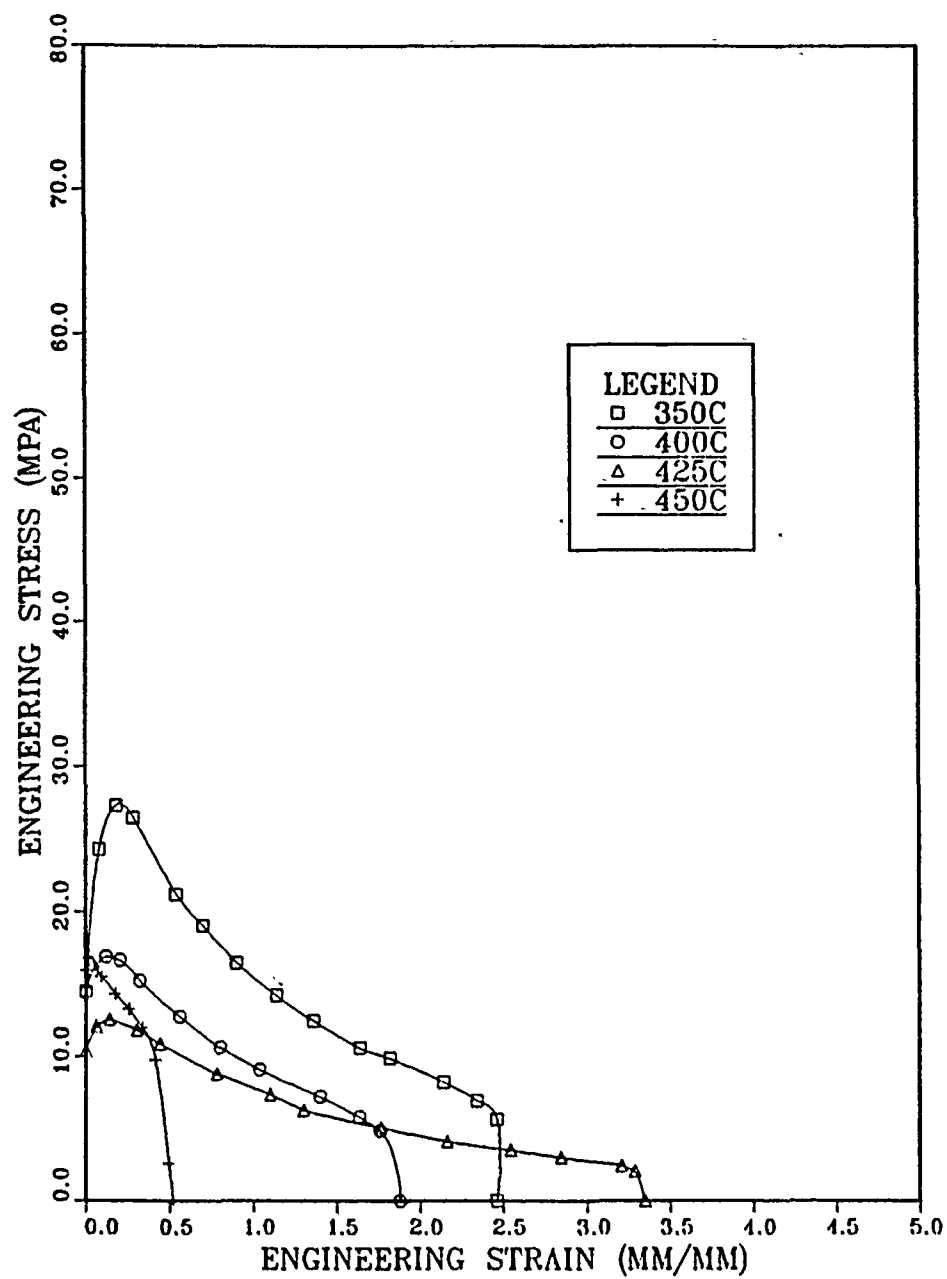


Figure 29. TMP E 2090 15min @ 300°C / 15min @ 250°C: Tested at $6.67 \times 10^{-4} \text{ sec}^{-1}$ strain rate

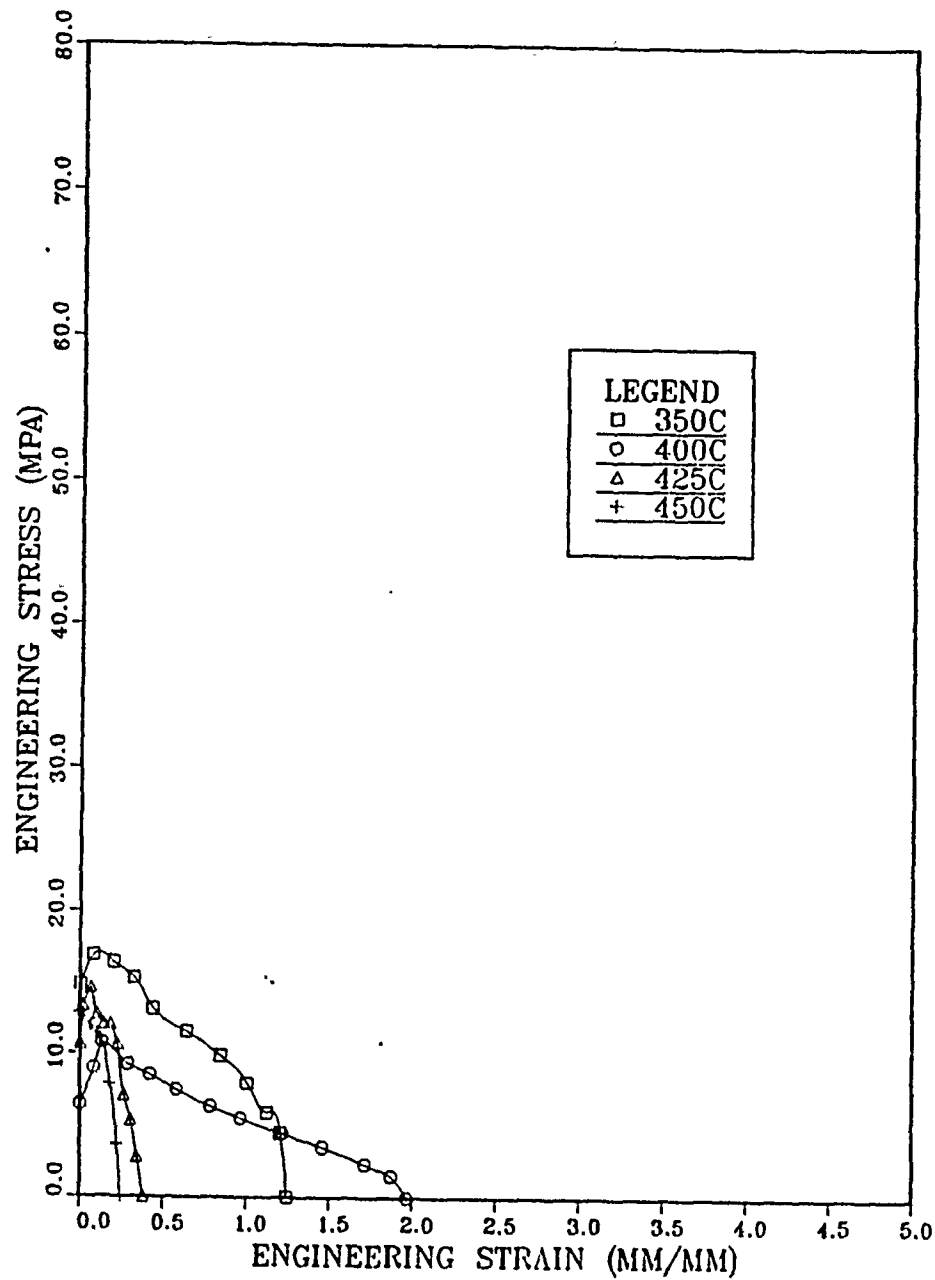


Figure 30. TMP E 2090 15min @ 300°C / 15min @ 250°C: Tested at $6.67 \times 10^{-3} \text{ sec}^{-1}$ strain rate

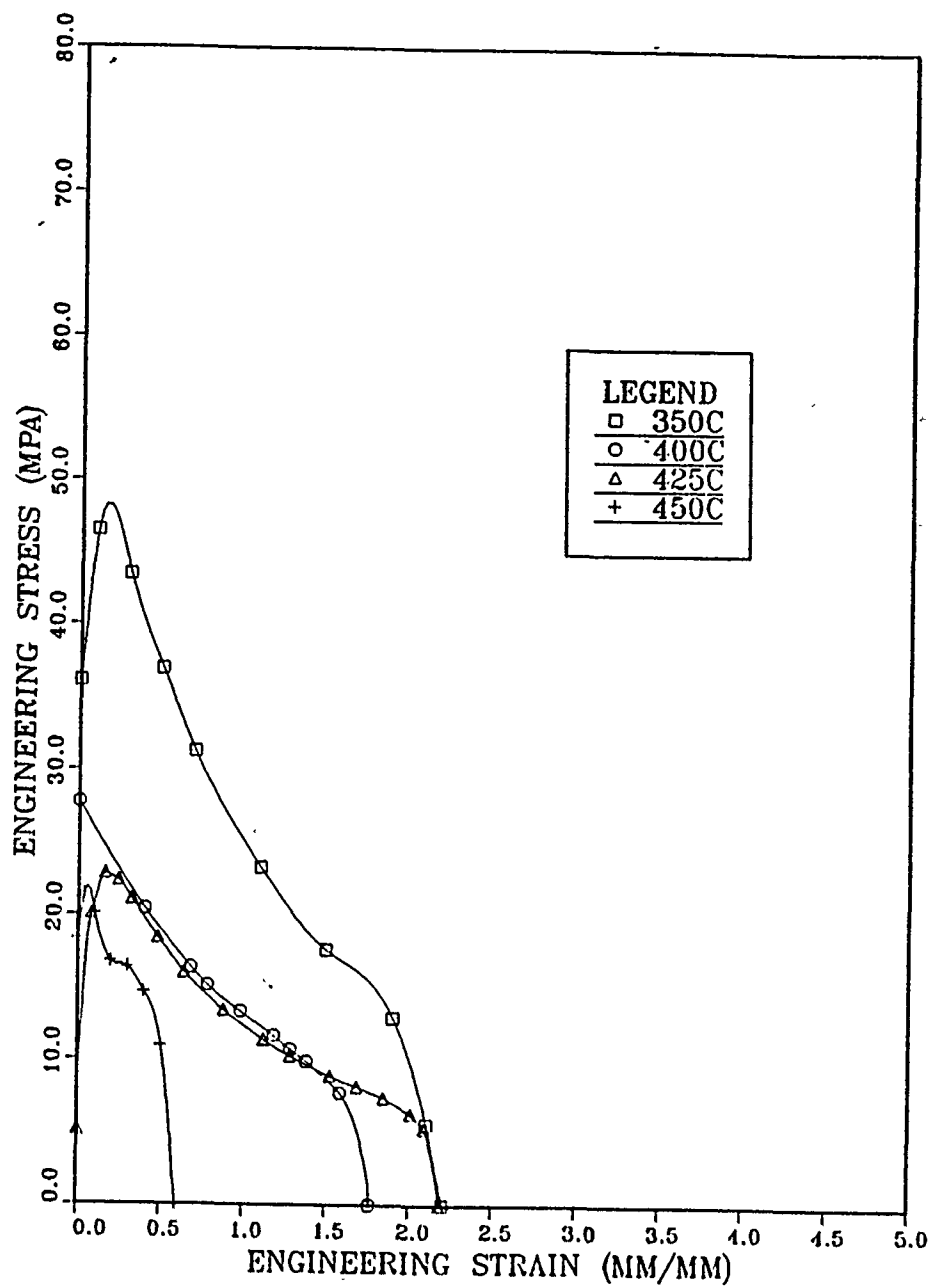


Figure 31. TMP F 2090 15min @ 300°C / 15min @ 200°C: Tested at $6.67 \times 10^{-5} \text{ sec}^{-1}$ strain rate

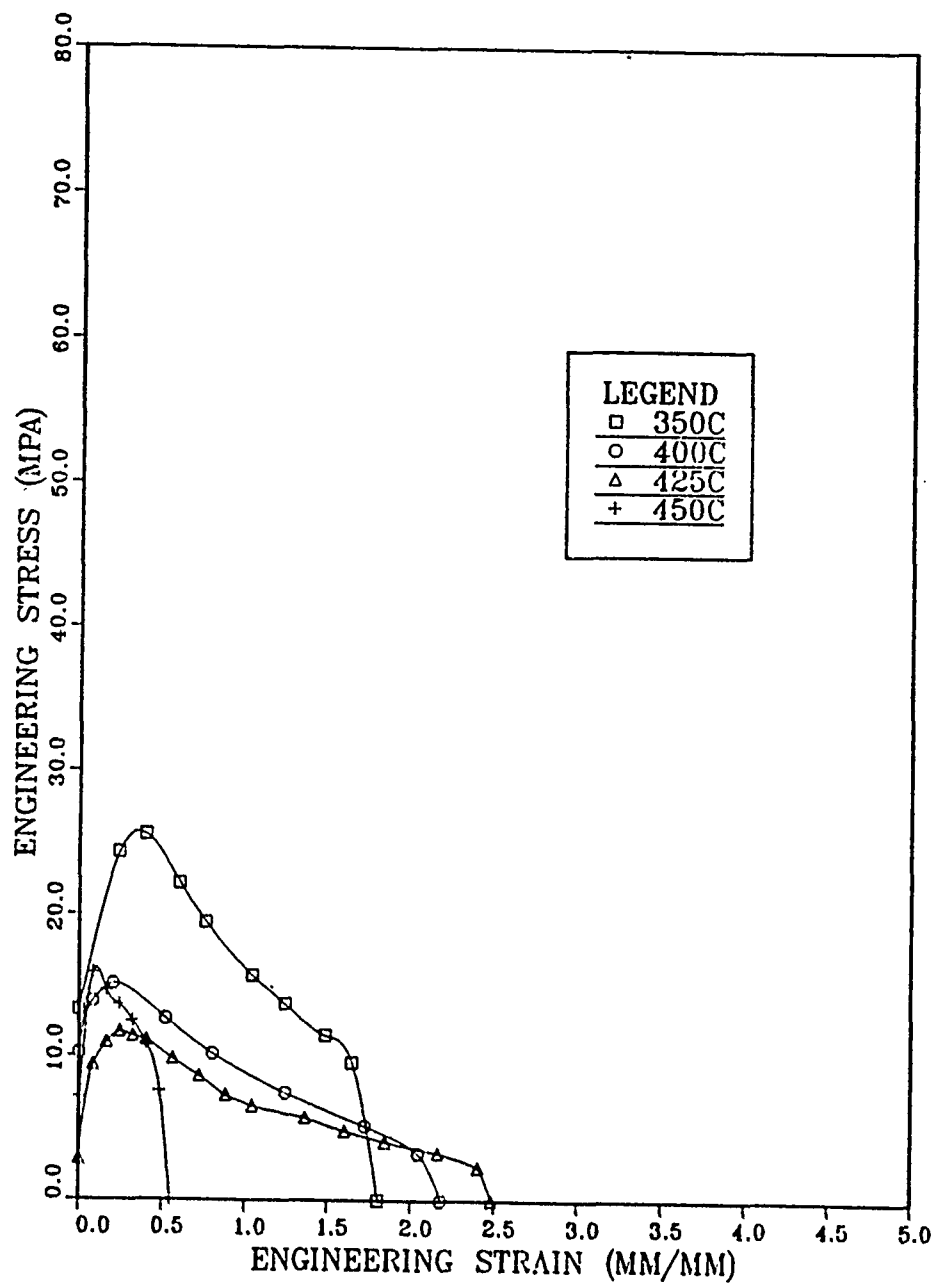


Figure 32. TMP F 2090 15min @ 300°C / 15min @ 200°C: Tested at $6.67 \times 10^{-4} \text{ sec}^{-1}$ strain rate

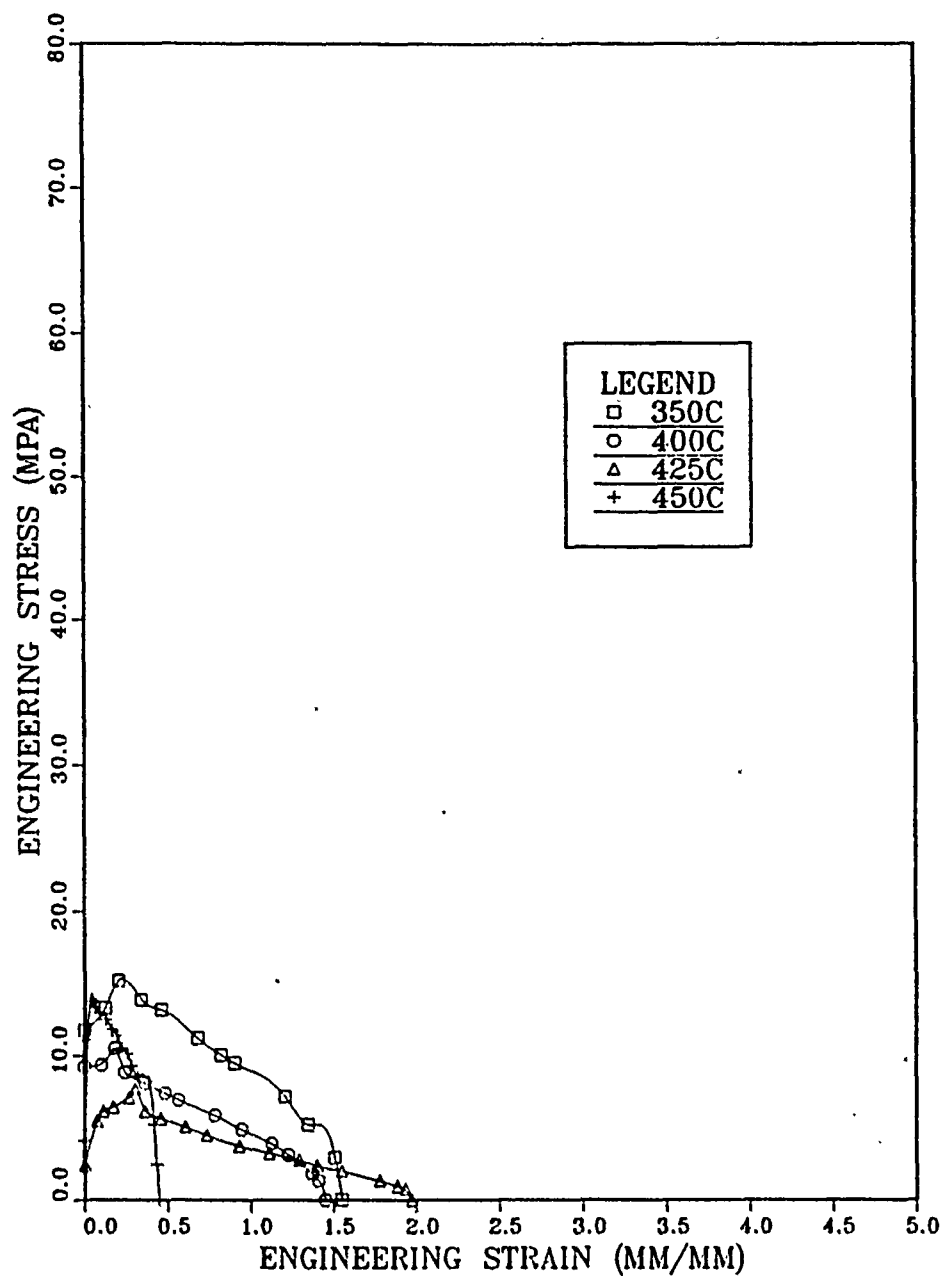


Figure 33. TMP F 2090 15min @ 300°C / 15min @ 200°C: Tested at $6.67 \times 10^{-3} \text{ sec}^{-1}$ strain rate

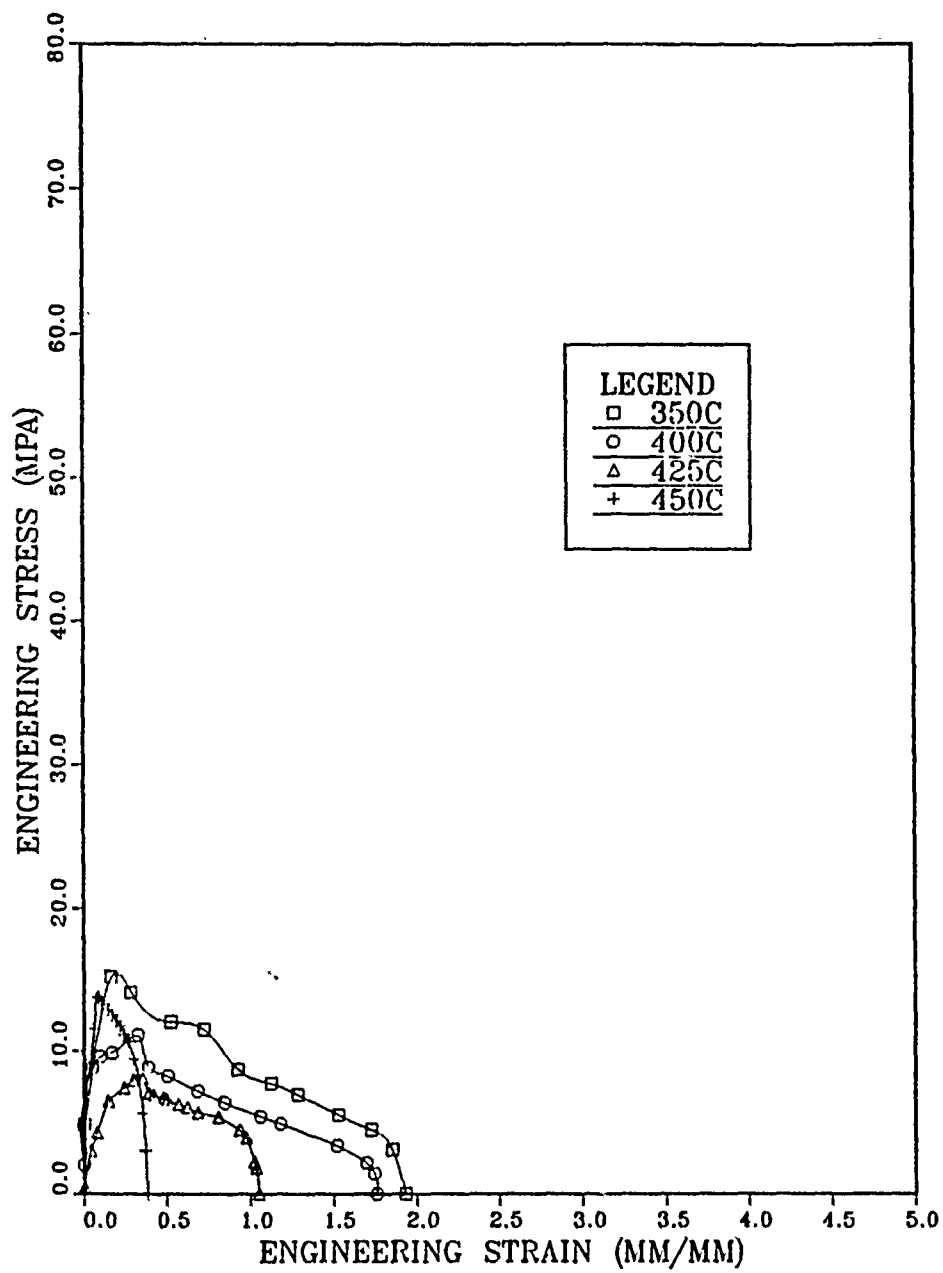


Figure 34. TMP G 2090 15min @ 300°C / 15min @ 150°C: Tested at $6.67 \times 10^{-5} \text{ sec}^{-1}$ strain rate

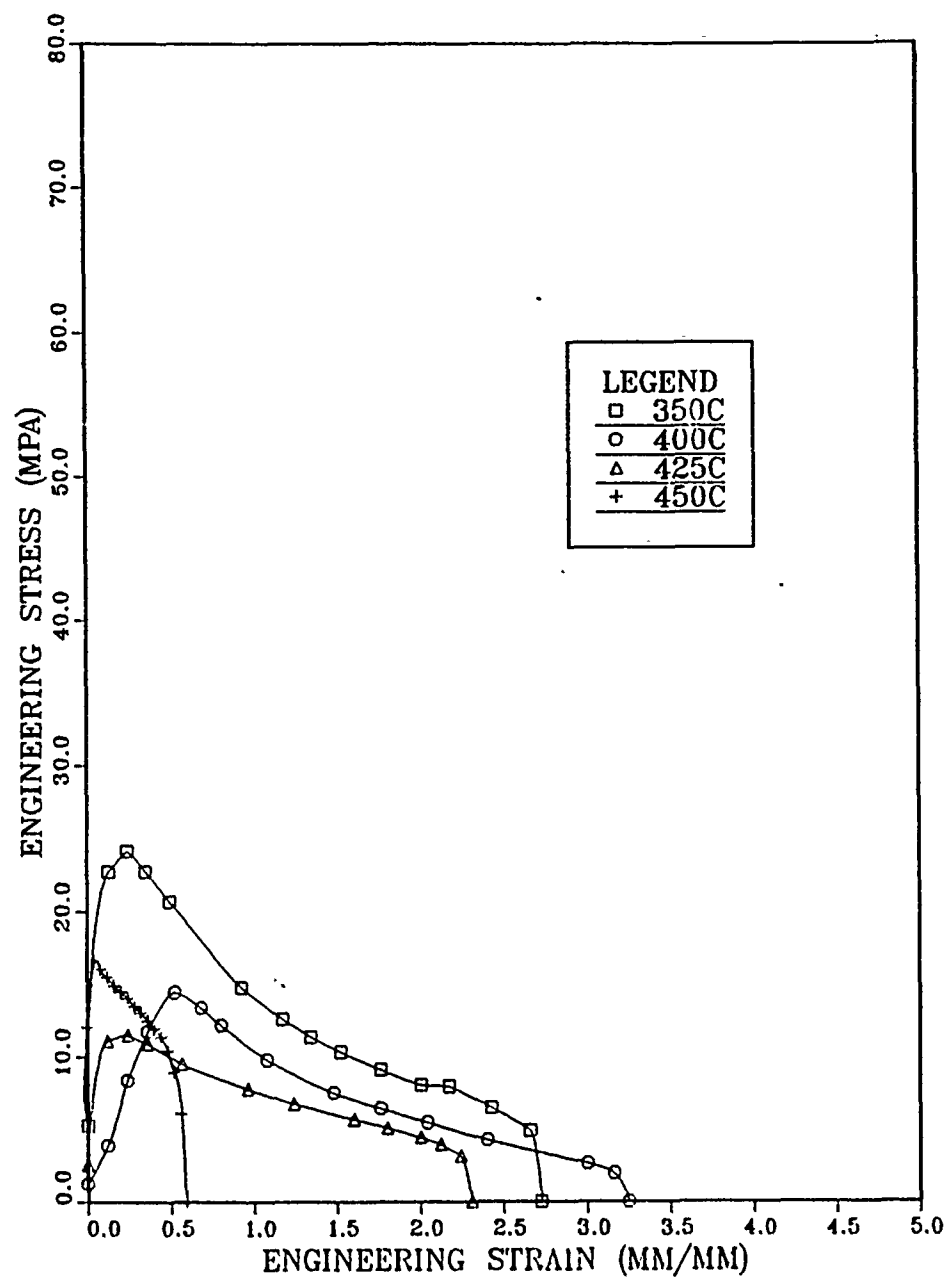


Figure 35. TMP G 2090 15min @ 300°C / 15min @ 150°C: Tested at $6.67 \times 10^{-4} \text{ sec}^{-1}$ strain rate

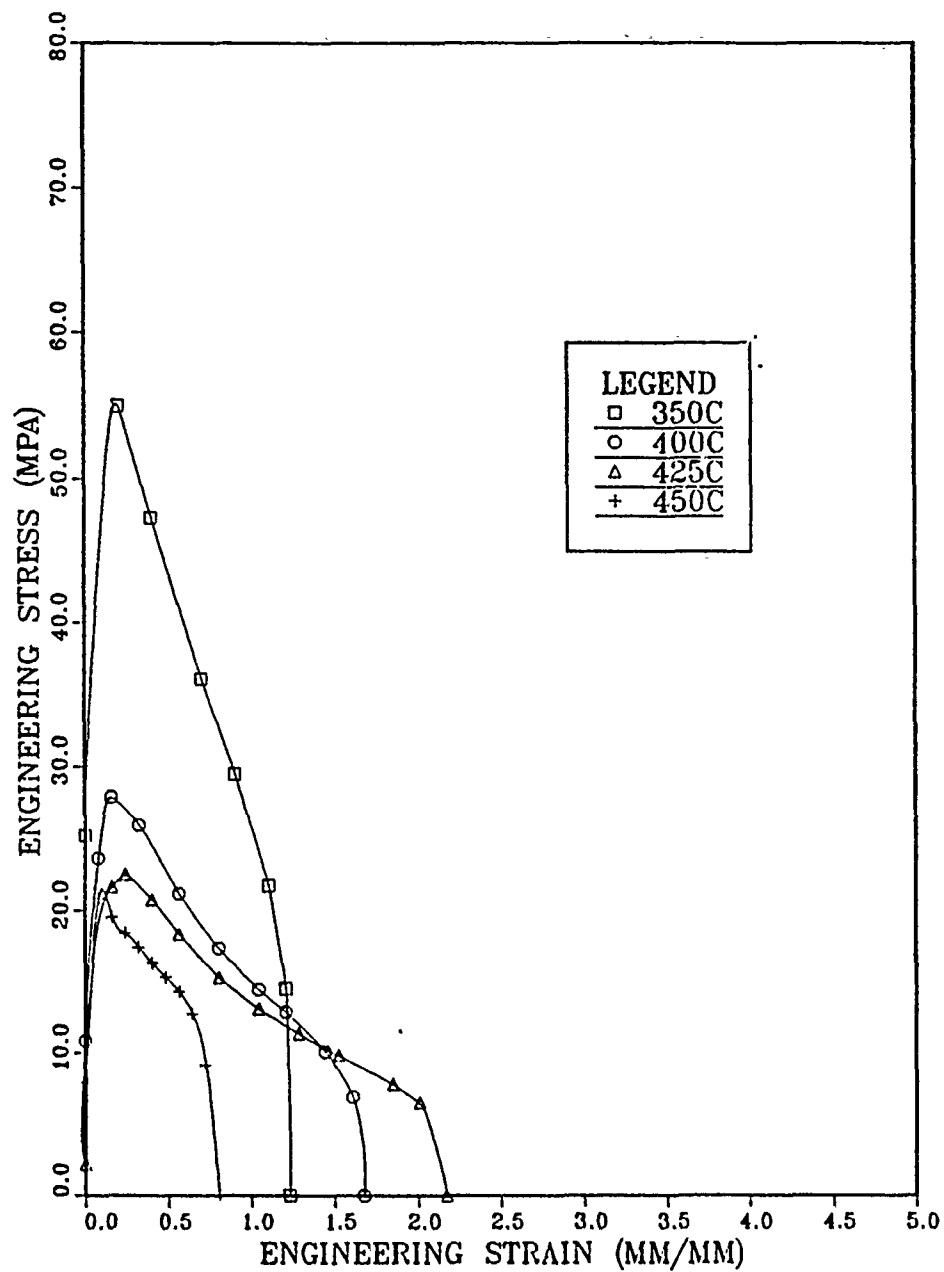


Figure 36. TMP G 2090 15min @ 300°C / 15min @ 150°C: Tested at $6.67 \times 10^{-3} \text{ sec}^{-1}$ strain rate

APPENDIX B. TRUE STRESS-STRAIN CURVES

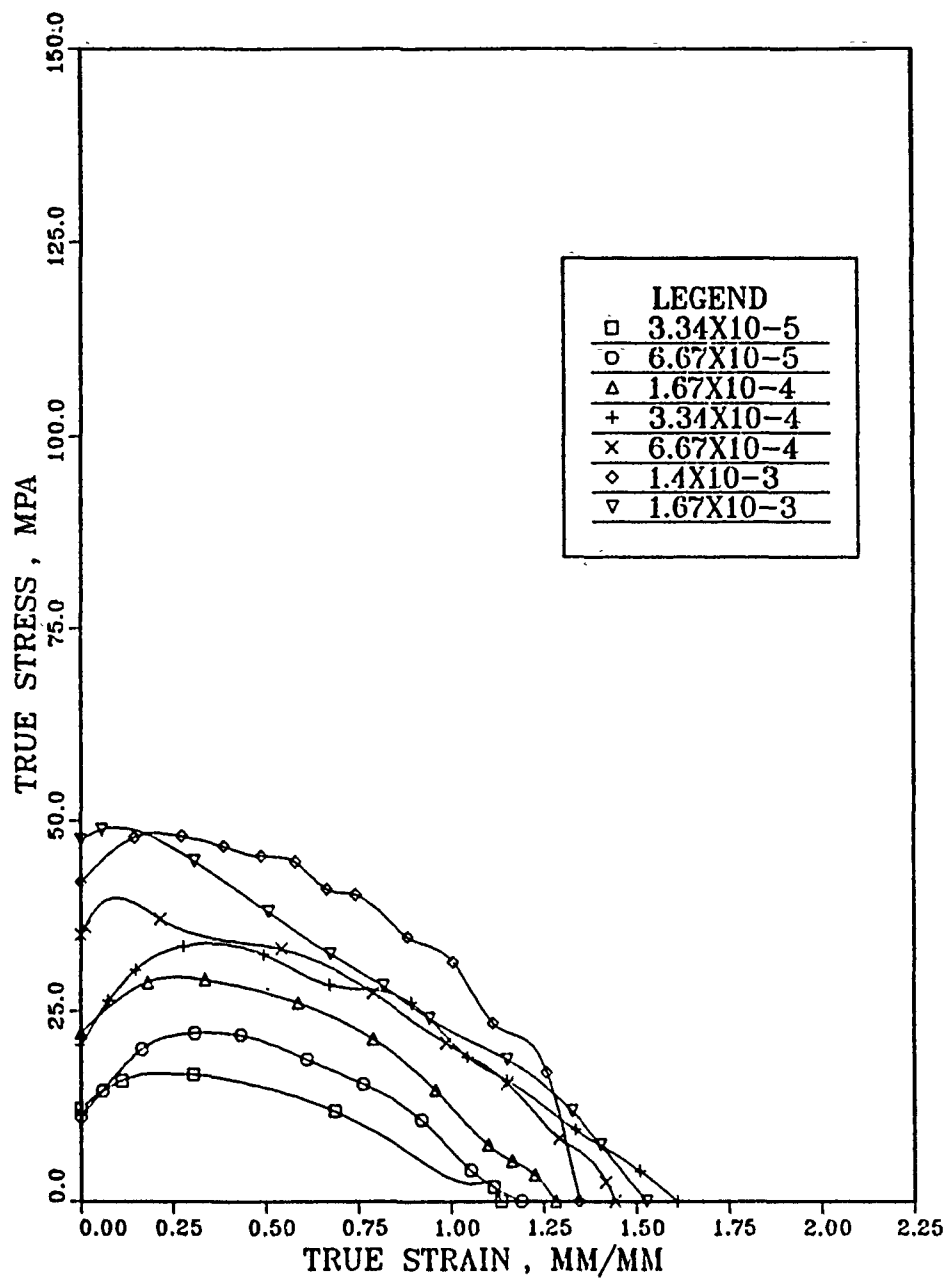


Figure 37. TMP A Al-10Mg-0.1Zr 60min @ 300°C

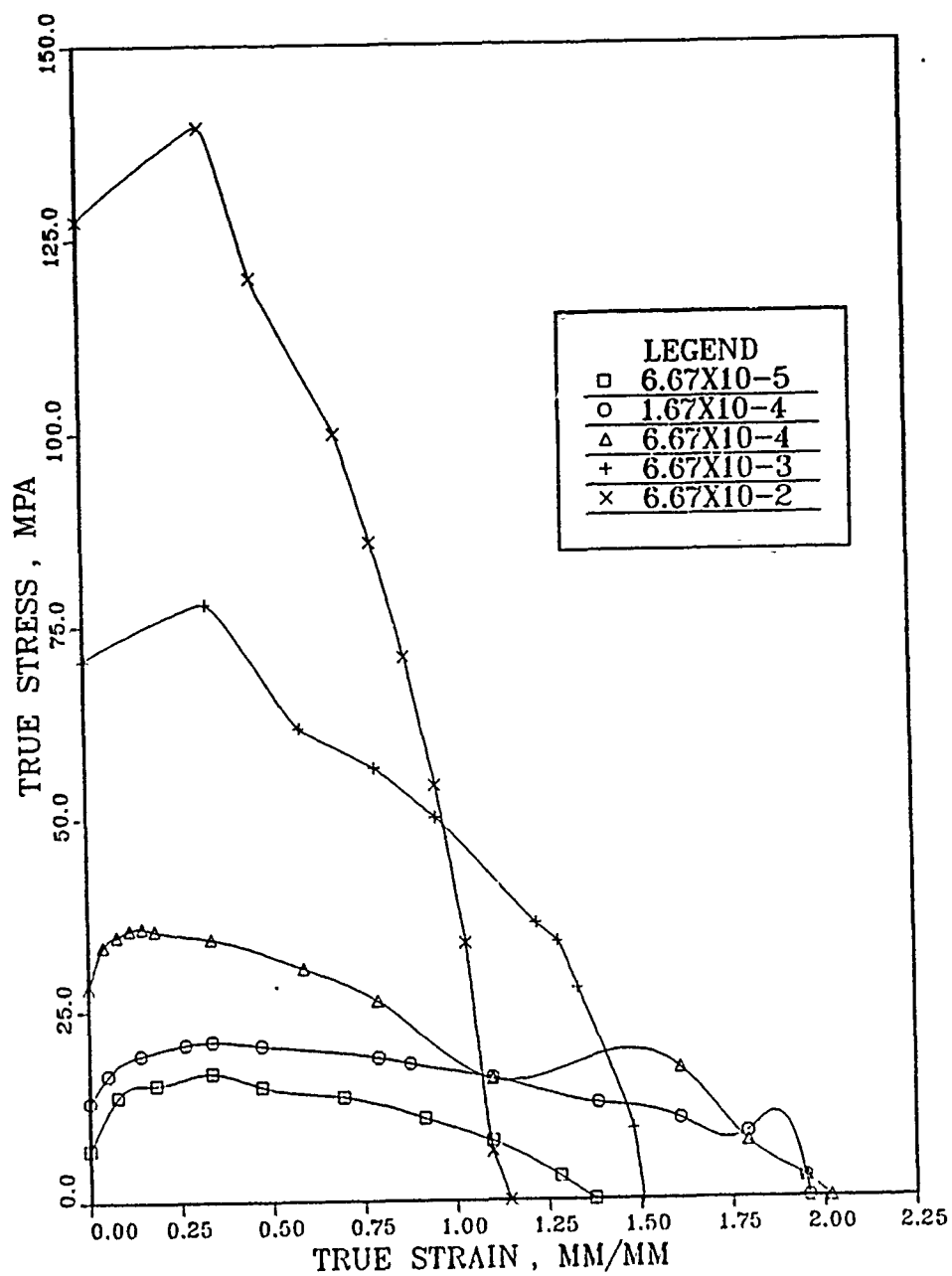


Figure 38. TMP B Al-10Mg-0.1Zr 30min @ 300°C / 15min @ 250°C

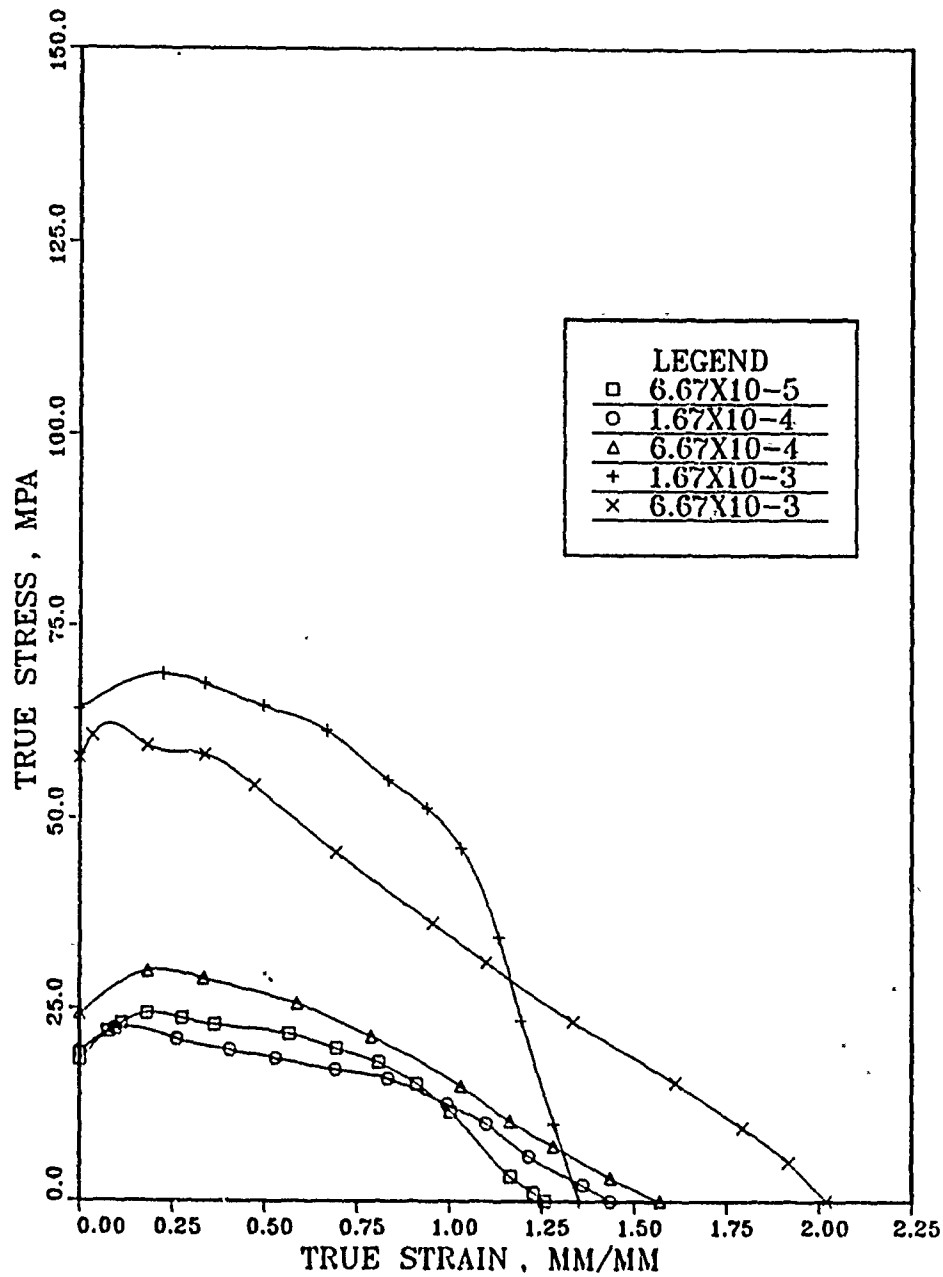


Figure 39. TMP C Al-10Mg-0.1Zr 30min @ 300°C / 15min @ 250°C

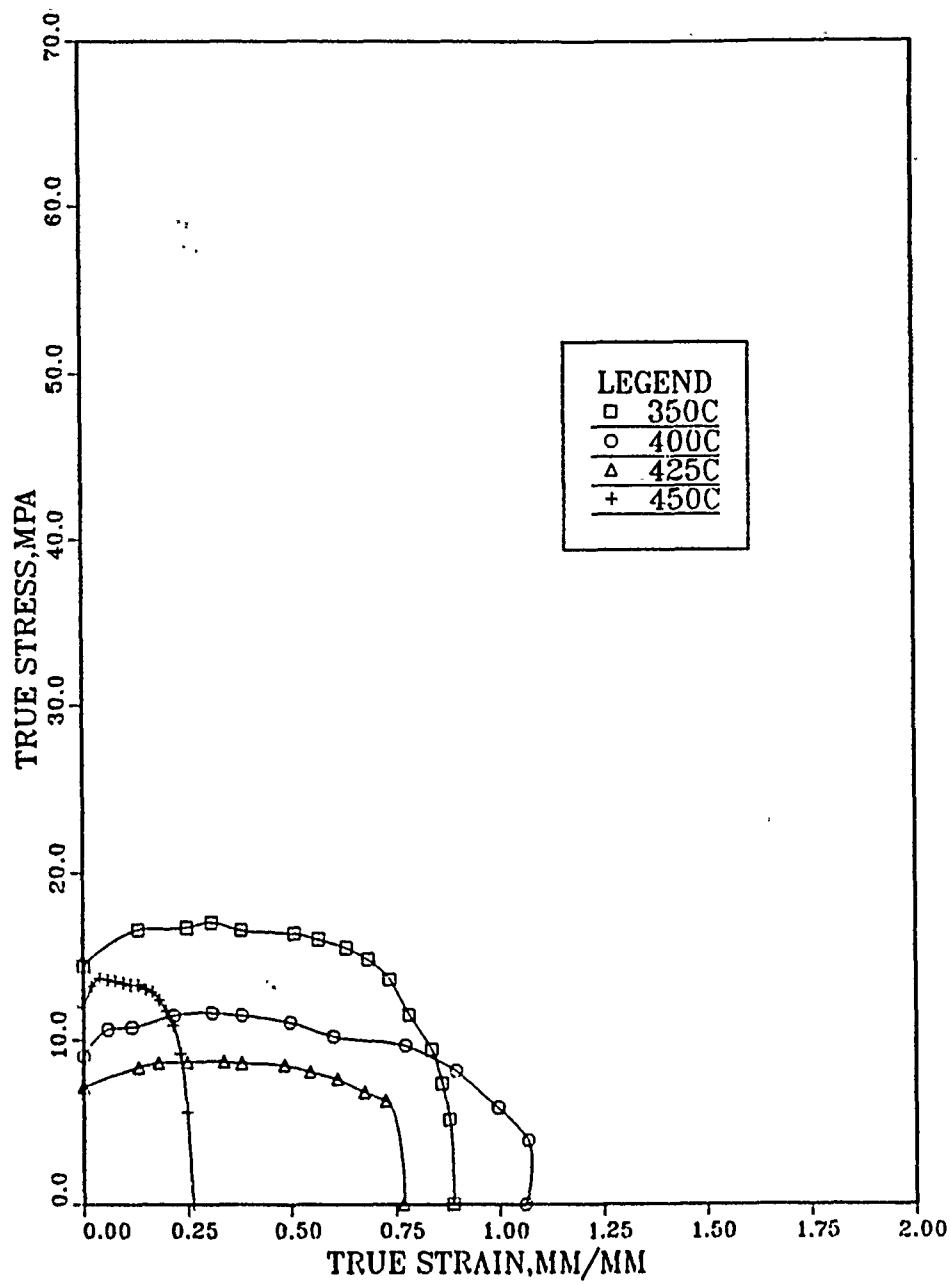


Figure 40. TMP D 2090 60min @ 300°C: Tested at $6.67 \times 10^{-5} \text{ sec}^{-1}$ strain rate

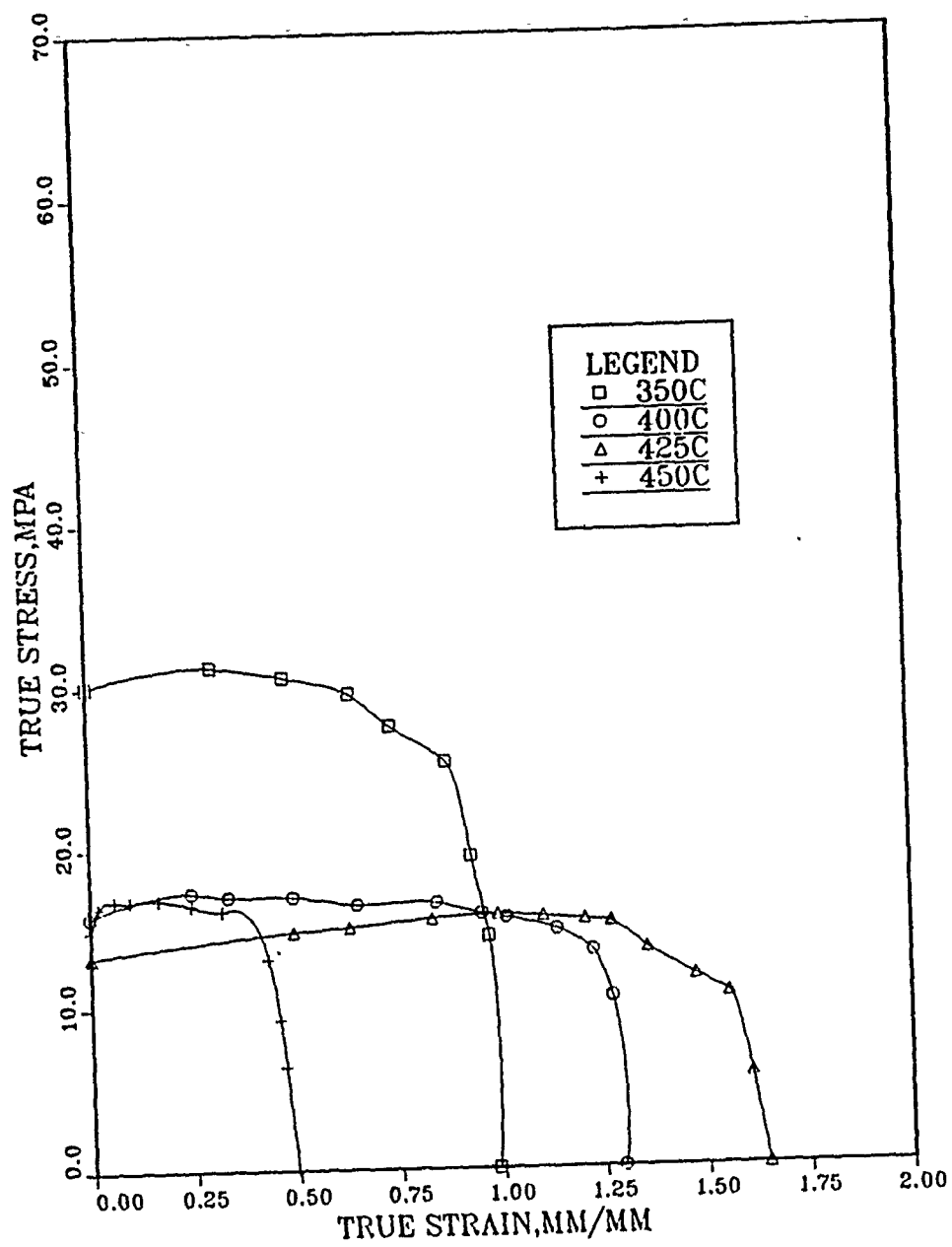


Figure 41. TMP D 2090 60min @ 300°C: Tested at $6.67 \times 10^{-4} \text{ sec}^{-1}$ strain rate

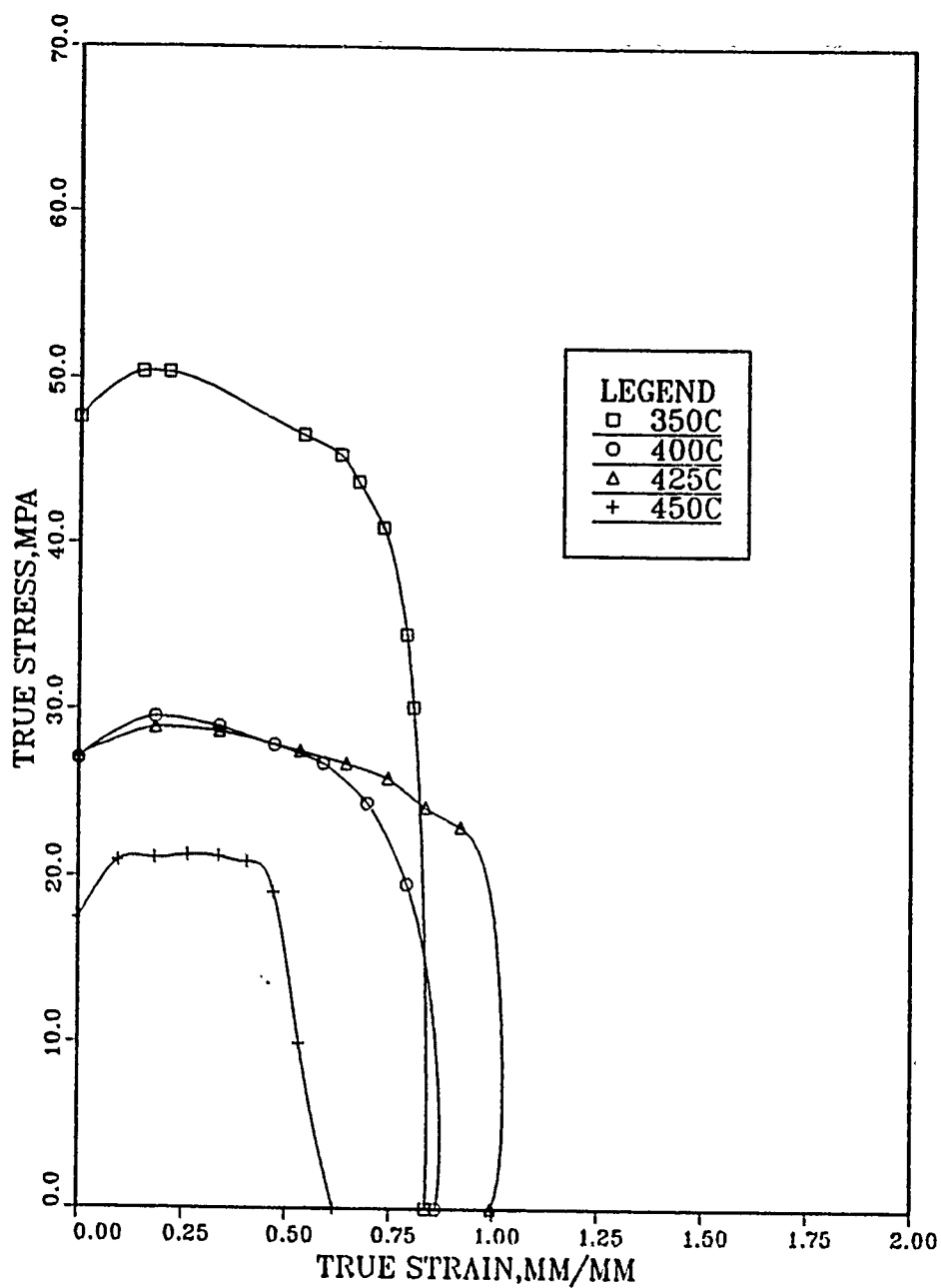


Figure 42. TMP D 2090 60min @ 300°C: Tested at $6.67 \times 10^{-3} \text{ sec}^{-1}$ strain rate

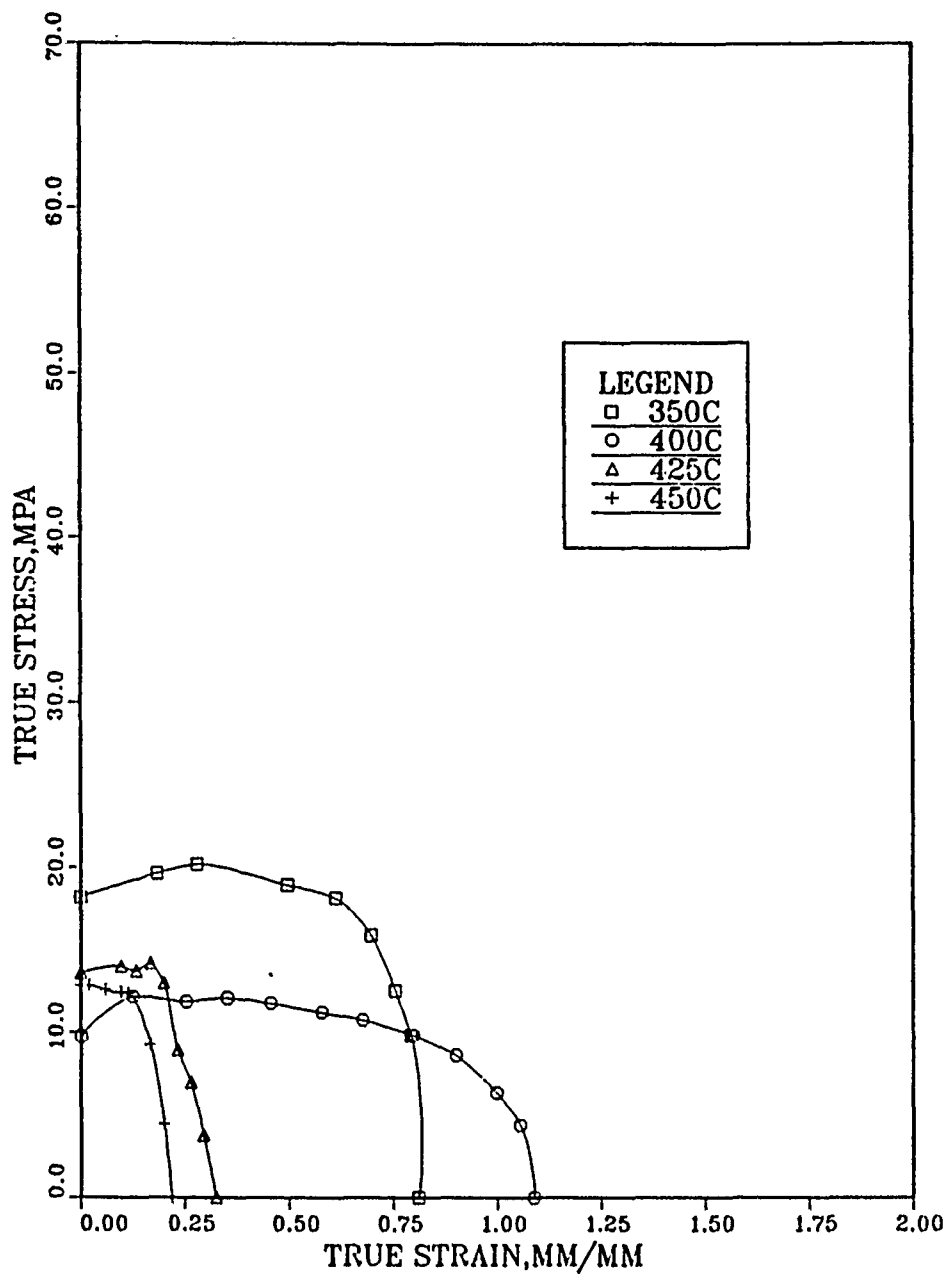


Figure 43. TMP E 2090 15min @ 300°C / 15min @ 250°C: Tested at $6.67 \times 10^{-5} \text{ sec}^{-1}$ strain rate

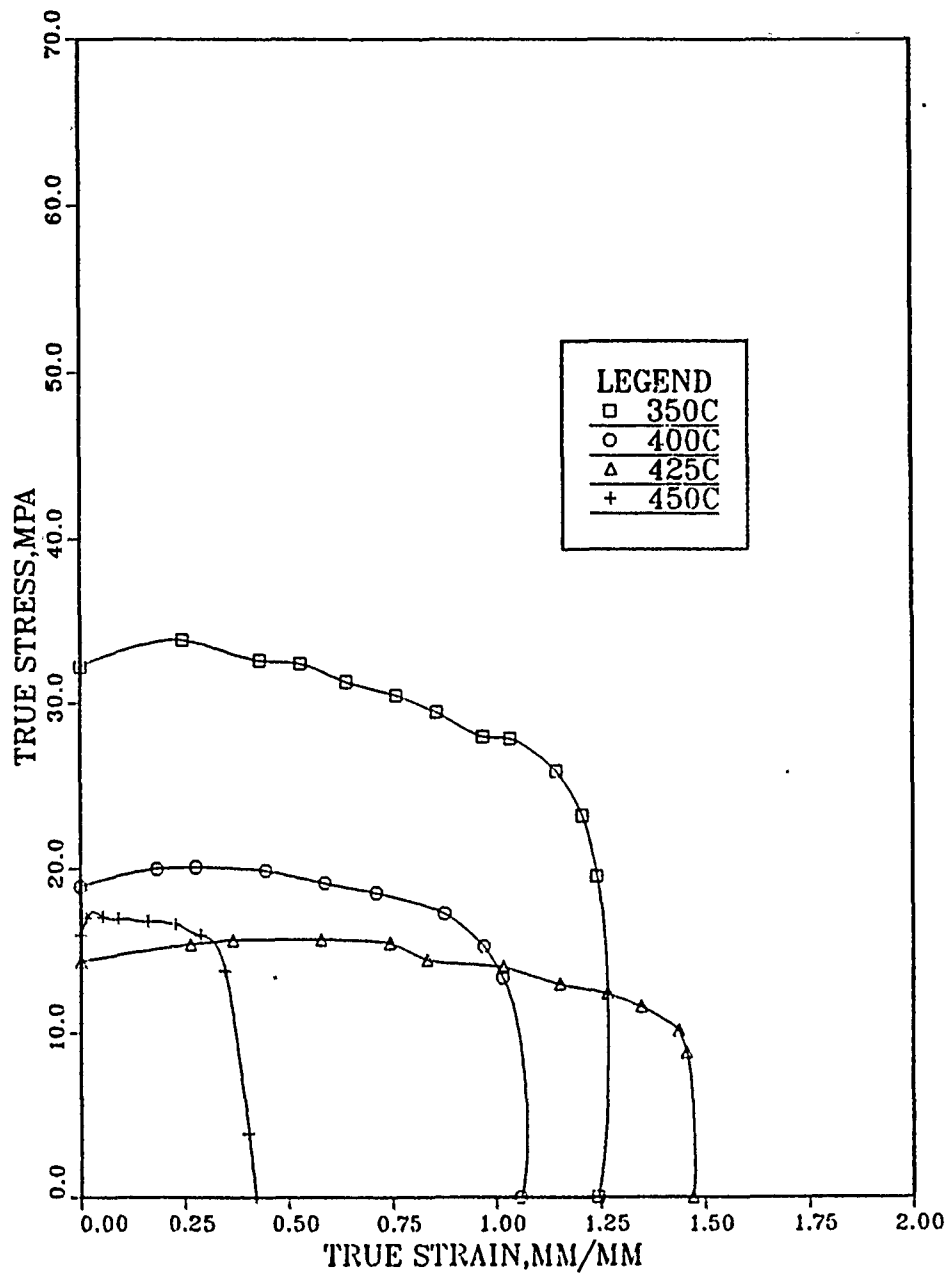


Figure 44. TMP E 2090 15min @ 300°C / 15min @ 250°C: Tested at $6.67 \times 10^{-4} \text{ sec}^{-1}$ strain rate

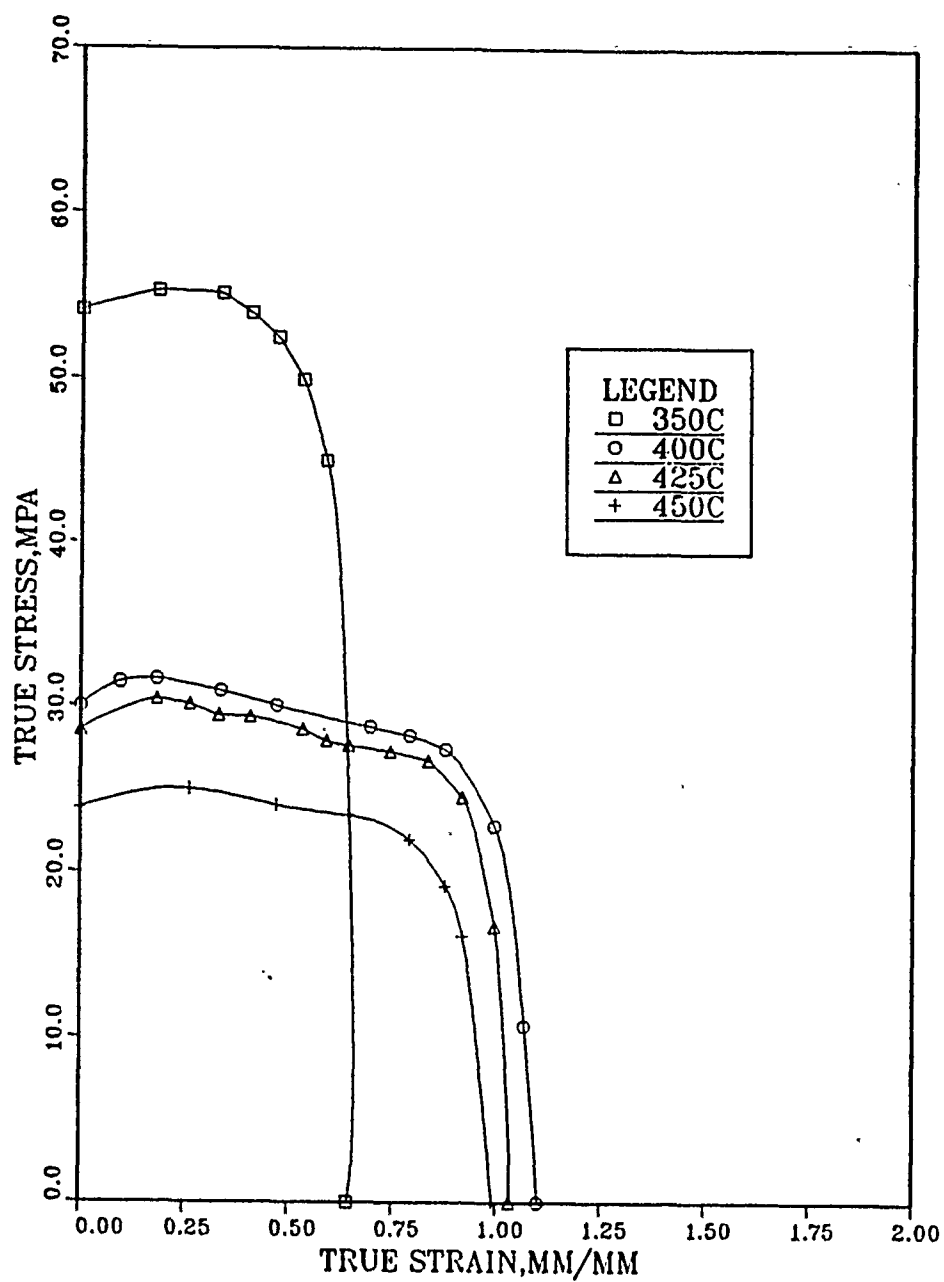


Figure 45. TMP E 2090 15min @ 300°C / 15min @ 250°C: Tested at $6.67 \times 10^{-3} \text{ sec}^{-1}$ strain rate

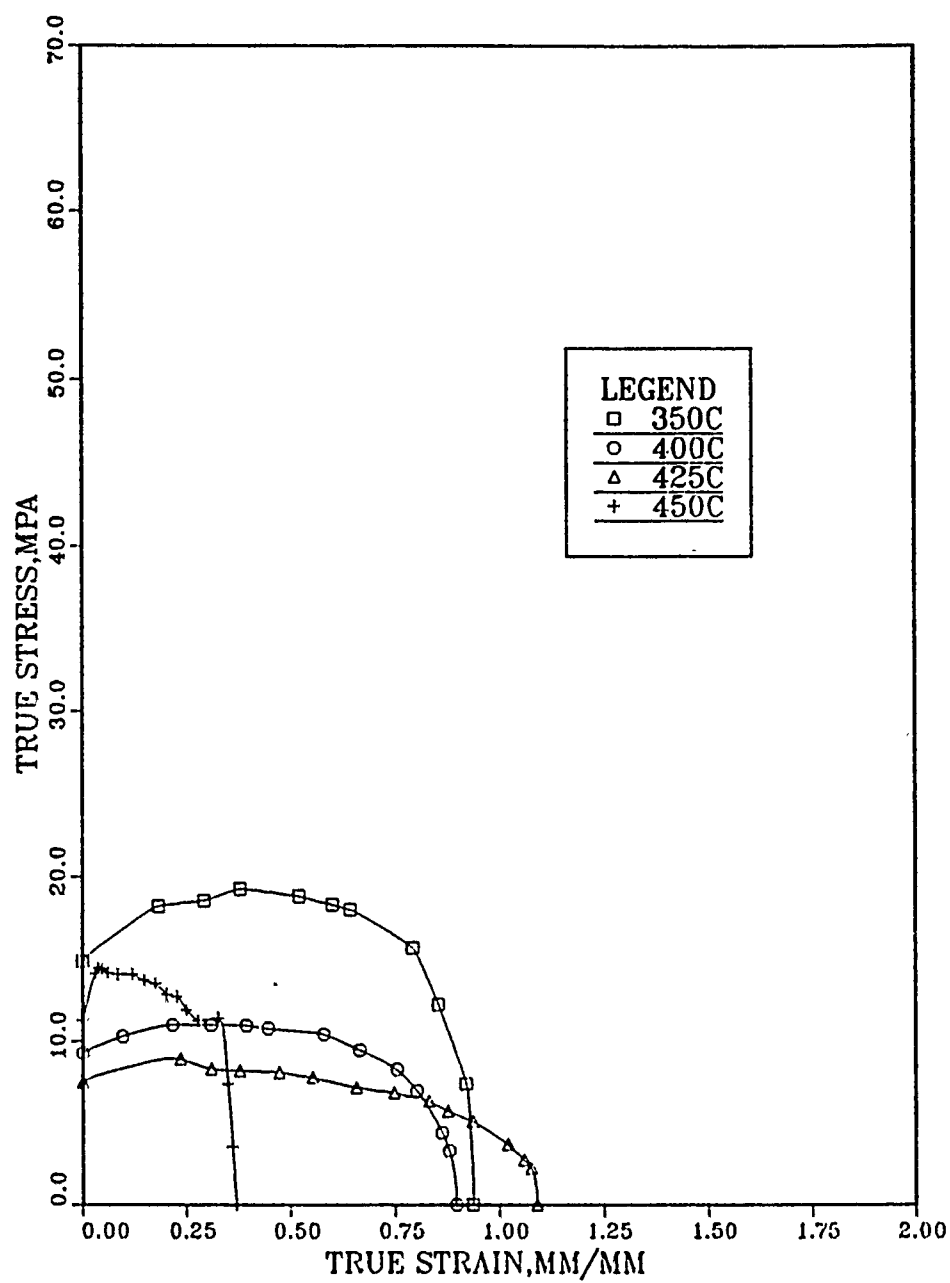


Figure 46. TMP F 2090 15min @ 300°C / 15min @ 200°C: Tested at $6.67 \times 10^{-4} \text{ sec}^{-1}$ strain rate

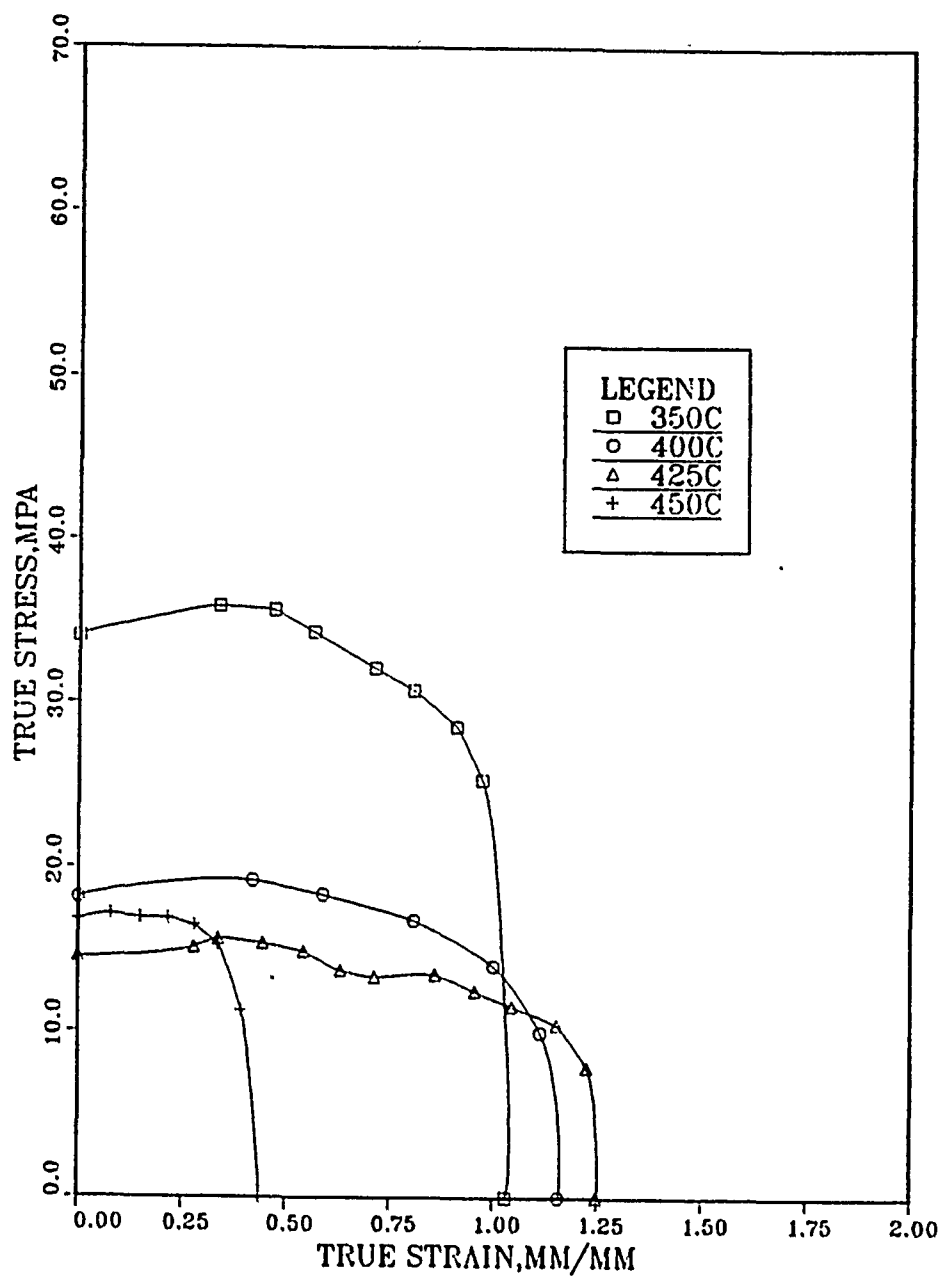


Figure 47. TMP F 2090 15min @ 300°C / 15min @ 200°C: Tested at $6.67 \times 10^{-4} \text{ sec}^{-1}$ strain rate

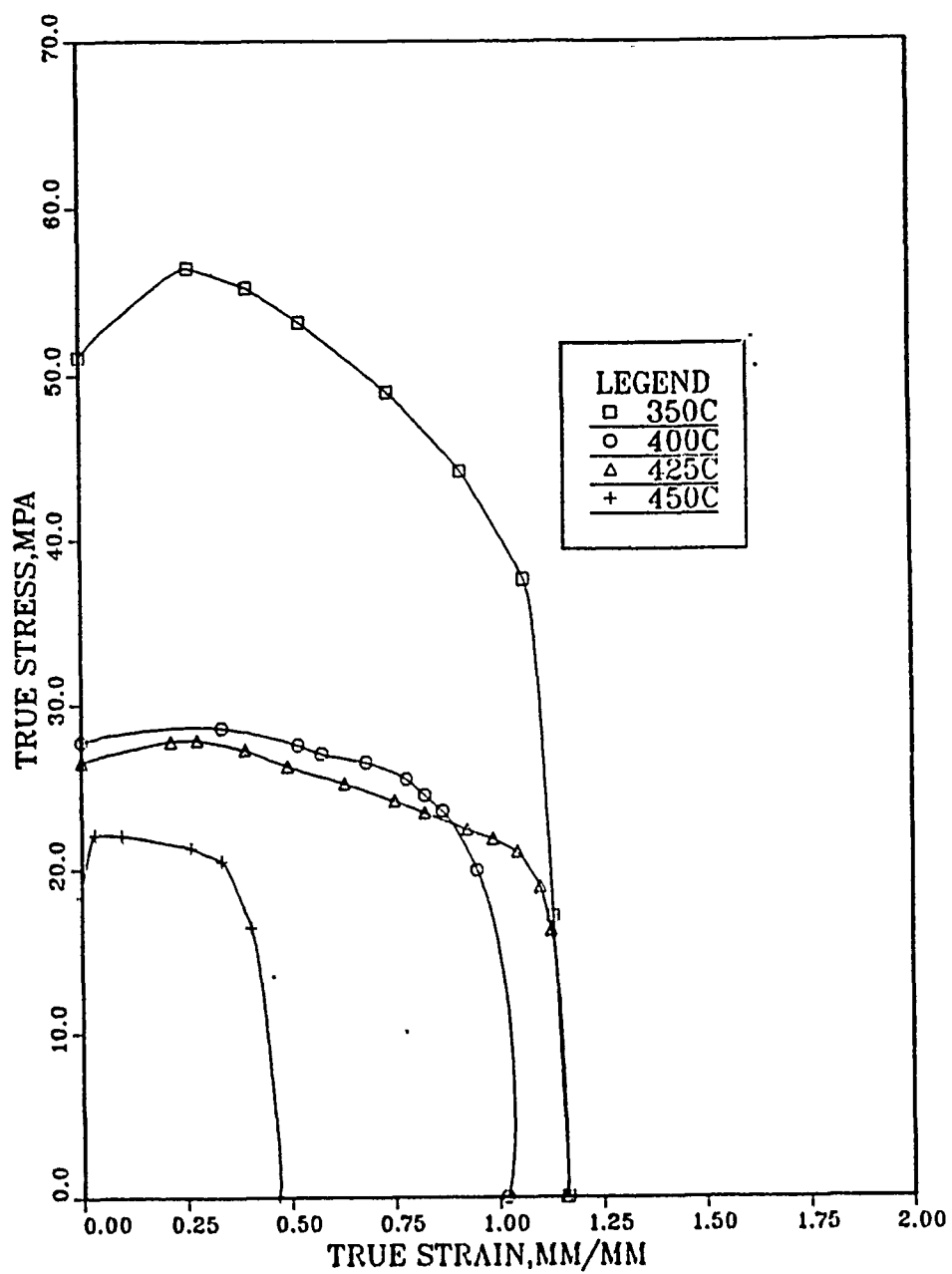


Figure 48. TMP F 2090 15min @ 300°C / 15min @ 200°C: Tested at $6.67 \times 10^{-3} \text{ sec}^{-1}$ strain rate

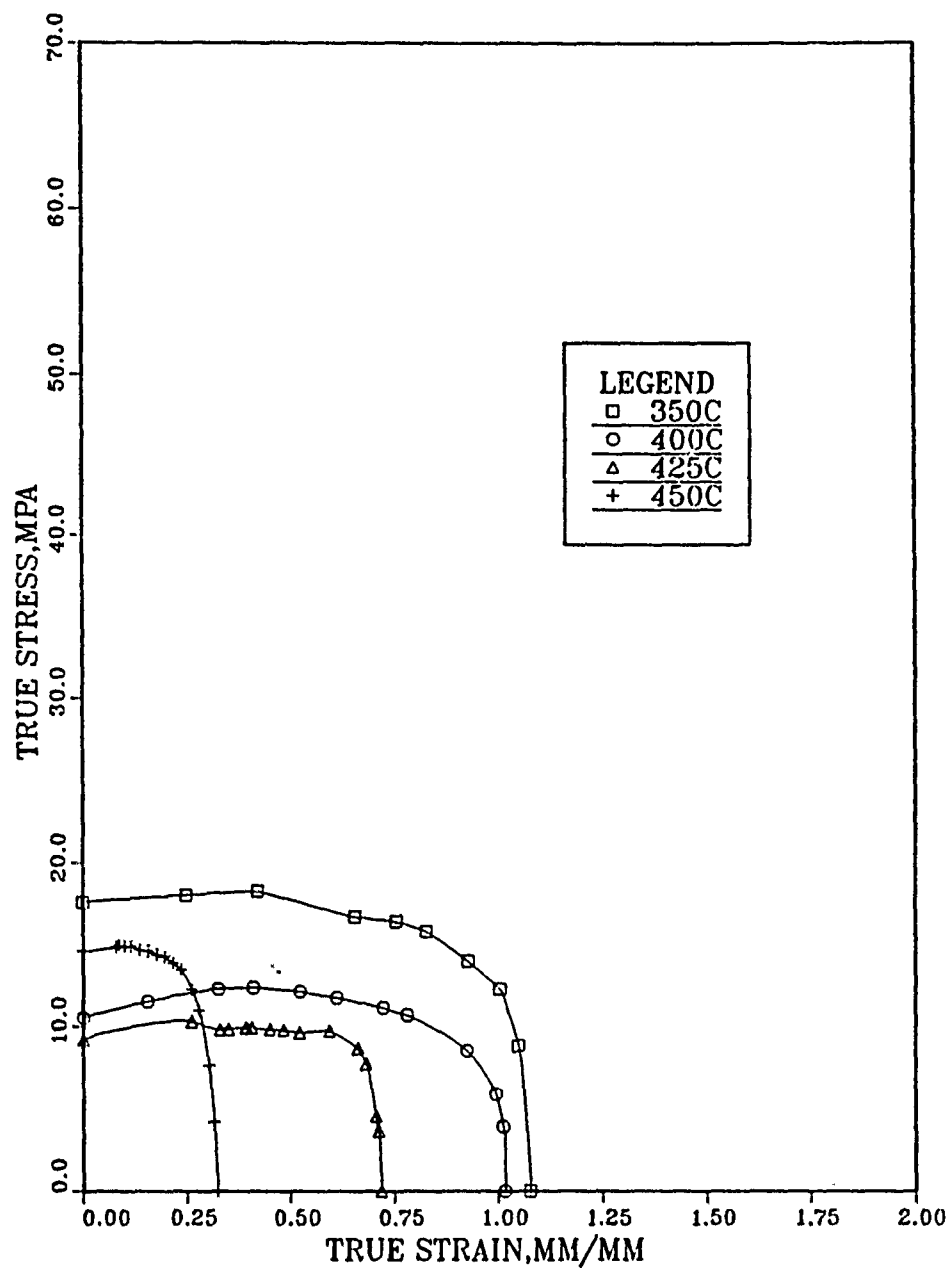


Figure 49. TMP G 2090 15min @ 300°C / 15min @ 150°C: Tested at $6.67 \times 10^{-5} \text{ sec}^{-1}$ strain rate

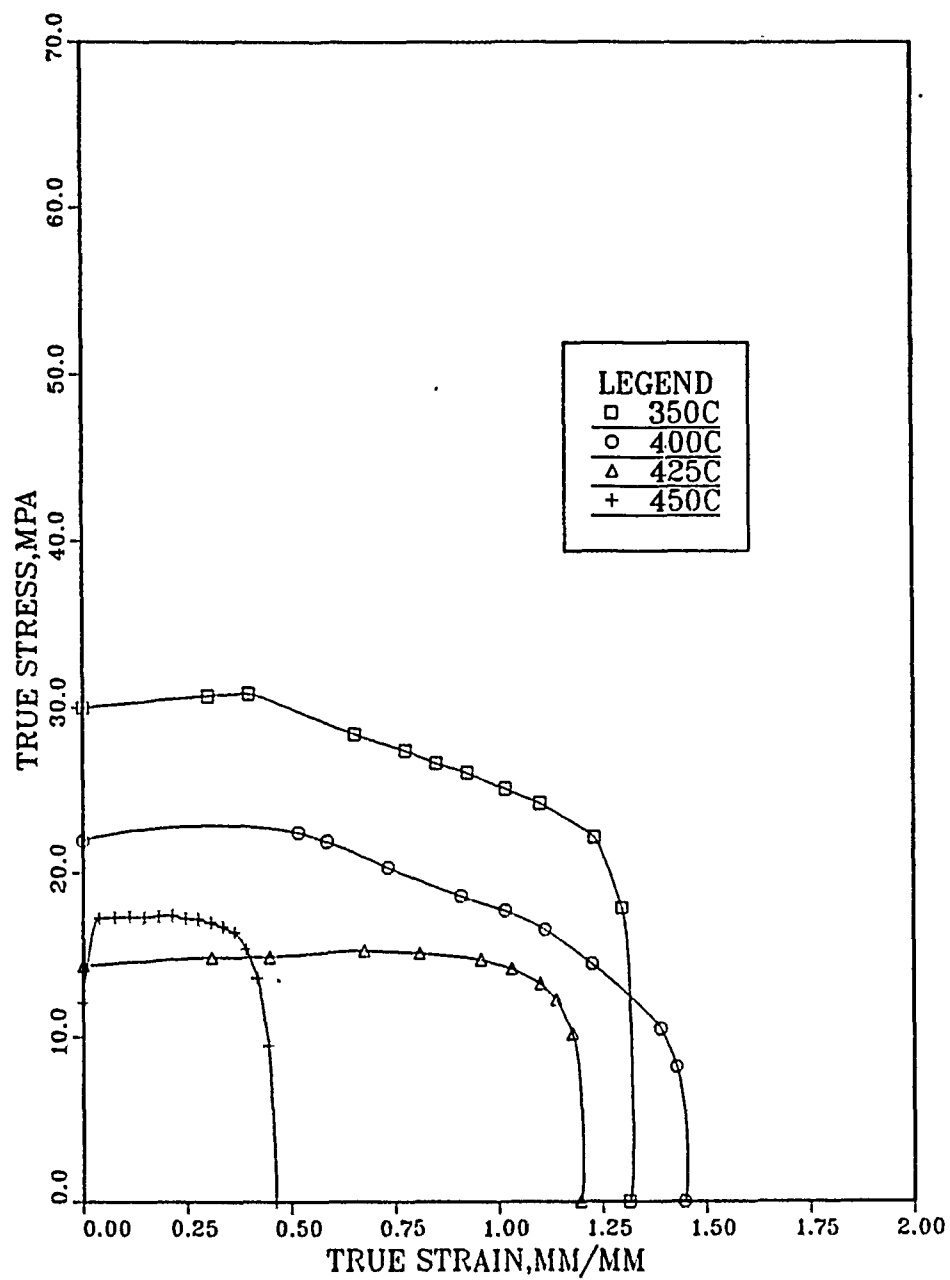


Figure 50. TMP G 2090 15min @ 300°C / 15min @ 150°C: Tested at $6.67 \times 10^{-4} \text{ sec}^{-1}$ strain rate

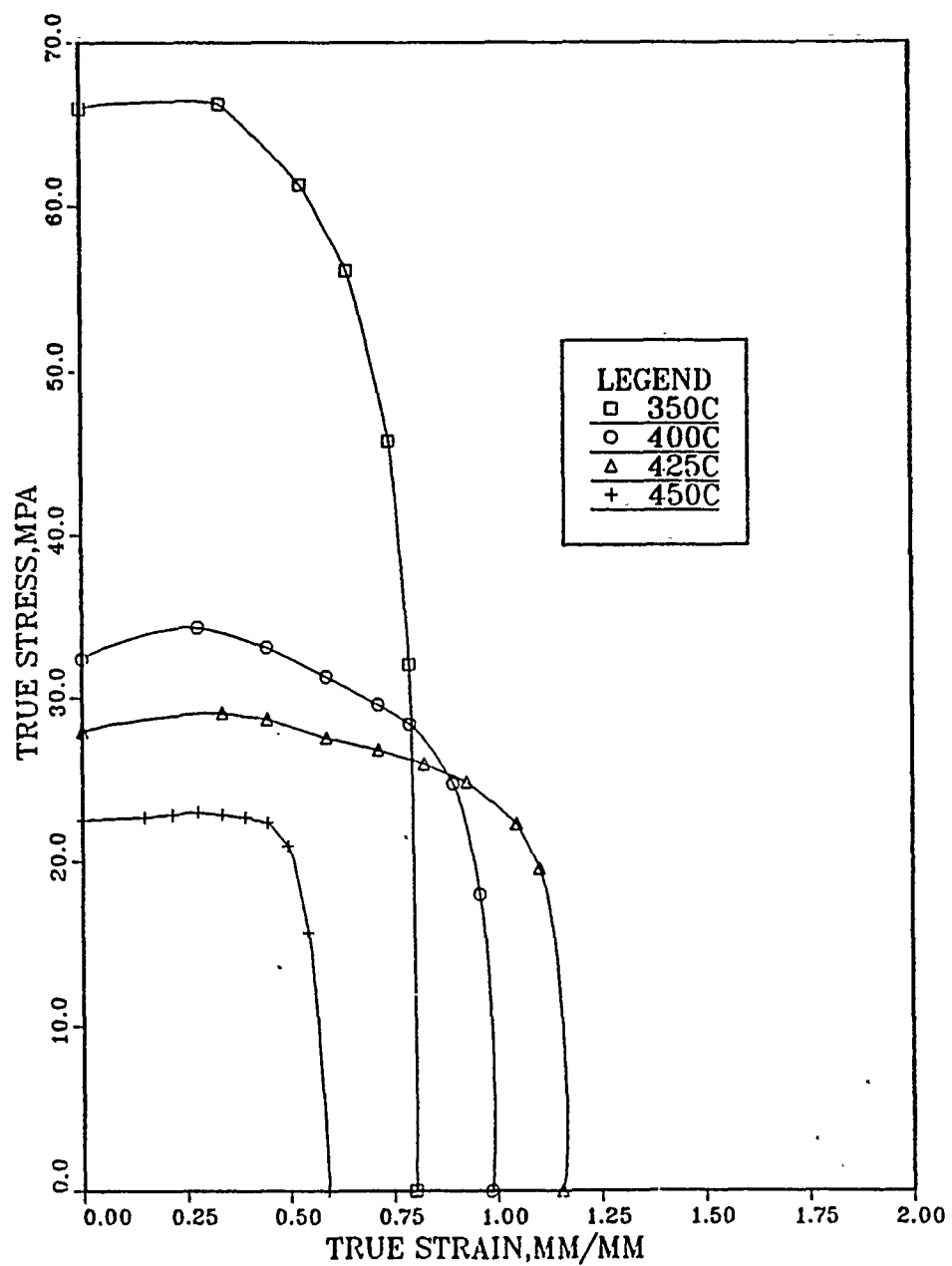


Figure 51. TMP G 2090 15min @ 300°C / 15min @ 150°C: Tested at $6.67 \times 10^{-3} \text{ sec}^{-1}$ strain rate

APPENDIX C. FLOW STRESS-STRAIN RATE CURVES

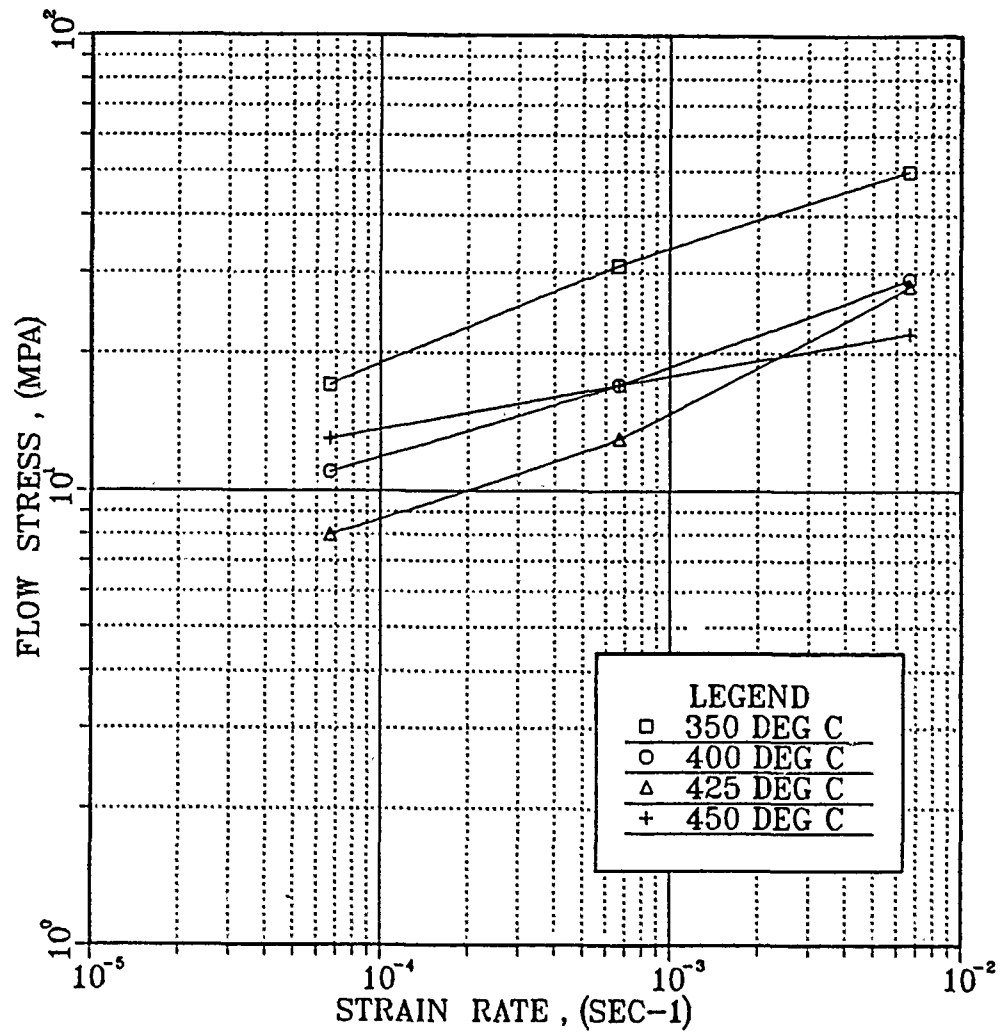


Figure 52. TMP D 2090 60min @ 300°C

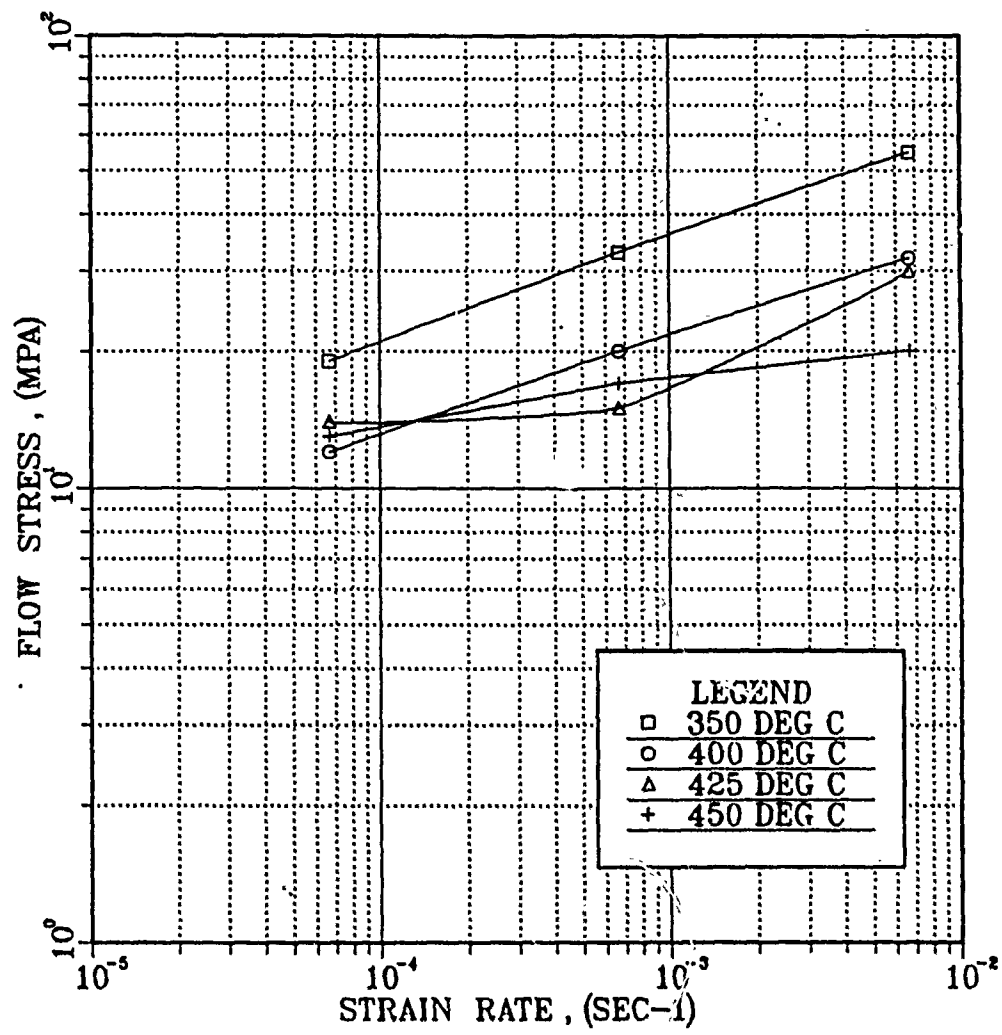


Figure 53. TMP E 2090 15min @ 300°C / 15min @ 250°C

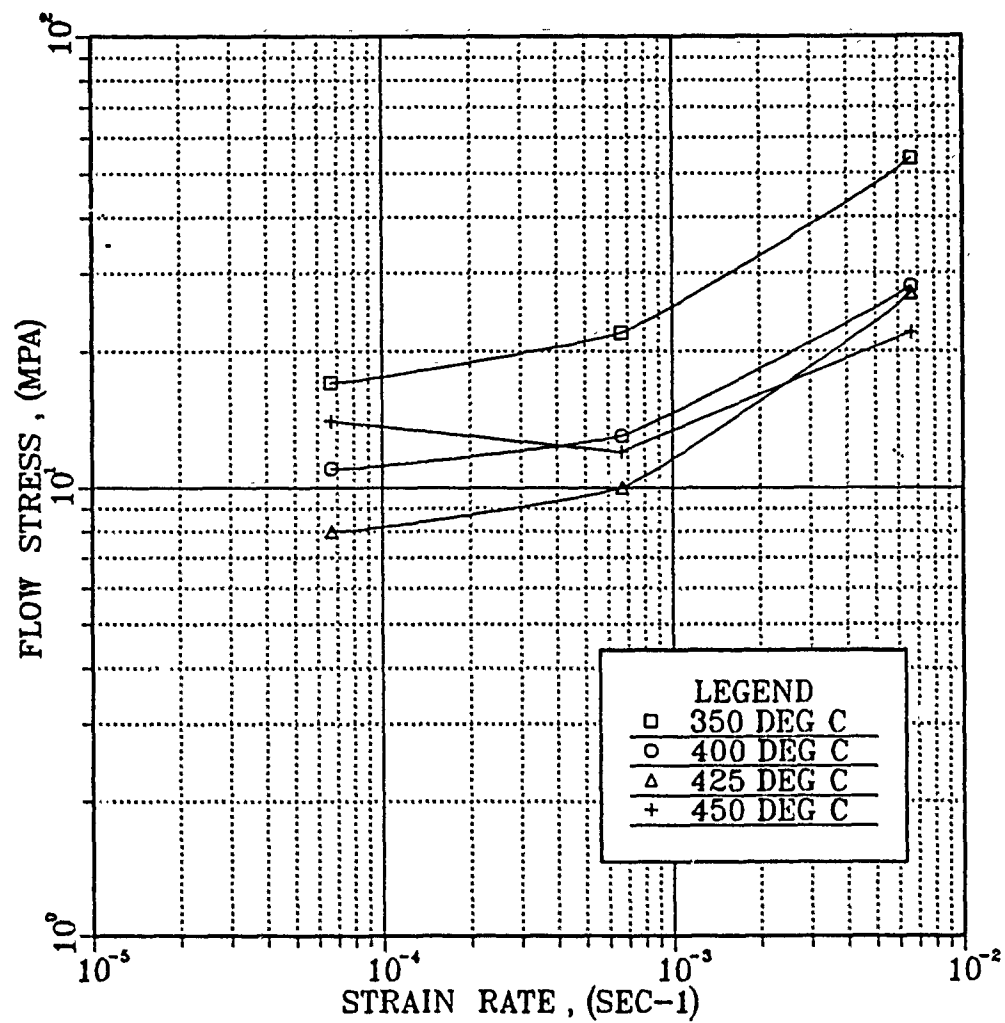


Figure 54. TMP F 2090 15min @ 300°C / 15min @ 200°C

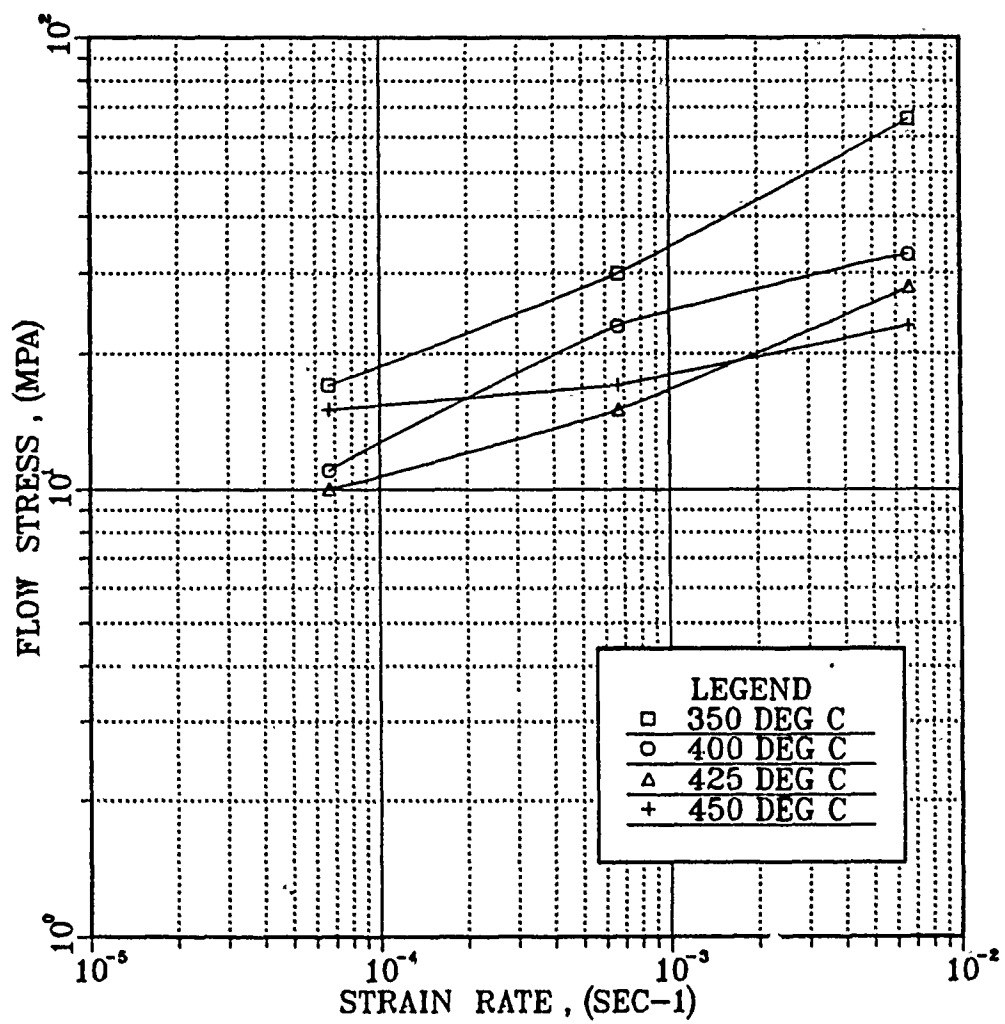


Figure 55. TMP G 2090 60min @ 300°C / 15min @ 150°C

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